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Cyclohexylammonium nitrate

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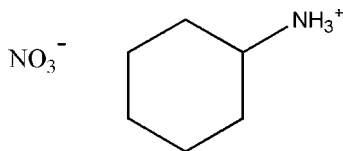
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.121; data-to-parameter ratio = 21.9.

In the title salt, $\text{C}_6\text{H}_{14}\text{N}^+\cdot\text{NO}_3^-$, the cyclohexyl ring adopts a chair conformation. The ammonium group occupies an equatorial position and the crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions, resulting in a three-dimensional network.

Related literature

For the Brønsted–Lowry basicity behavior of cyclohexylamine, see: Solomons (1996). For the preparation of salts of anions and complex anions with cyclohexyl primary ammonium cations, see: Jones *et al.* (1998); Kolev *et al.* (2007); Lock *et al.* (1981); Muthamizhchelvan *et al.* (2005); Wang *et al.* (2005); Yun *et al.* (2004). For precautions relating to the reaction of cyclohexylamine with strong acids or oxidizing agents, see: Chang (2008); Patnaik (2007). For the structures of other cyclohexyleammonium salts, see: Shimada *et al.* (1955); Smith *et al.* (1994); Odendal *et al.* (2010). For ring conformations and ring puckering analysis, see: Cremer & Pople (1975). For reference bond lengths, see: Allen *et al.* (1987).



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¶ Thomson Reuters ResearcherID: A-3561-2009.

Experimental

Crystal data

$\text{C}_6\text{H}_{14}\text{N}^+\cdot\text{NO}_3^-$
 $M_r = 162.19$
 Monoclinic, $P2_1/c$
 $a = 8.9322$ (9) Å
 $b = 9.9010$ (9) Å
 $c = 10.3951$ (10) Å
 $\beta = 103.866$ (2)°
 $V = 892.53$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 294$ K
 $0.39 \times 0.15 \times 0.14$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.964$, $T_{\max} = 0.987$
 2214 measured reflections
 2214 independent reflections
 1750 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.121$
 $S = 1.09$
 2214 reflections
 101 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.97	1.89	2.8553 (14)	172
$\text{N1}-\text{H2}\cdots\text{O3}^{\text{ii}}$	0.94	1.97	2.9074 (15)	172
$\text{N1}-\text{H3}\cdots\text{O1}^{\text{iii}}$	0.85	2.24	2.9880 (15)	148
$\text{N1}-\text{H3}\cdots\text{O3}^{\text{iii}}$	0.85	2.28	3.0689 (15)	155
Symmetry codes:	(i) $-x+2, -y+1, -z+2$;	(ii) $x, y-1, z$;	(iii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$;	

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SJ5386).

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supporting information

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Cyclohexylammonium nitrate

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S1. Comment

The title compound $C_6H_{11}NH_3^+NO_3^-$ was obtained as the unexpected by-product of the reaction of metal (M) nitrate salts (metal = Mg^{2+} , Al^{3+} , Cr^{3+} , Mn^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} , or Cd^{2+}) with cyclohexylamine (CHA) in either aqueous or ethanolic media. It was expected that CHA would coordinate to the M cations due to its Lewis basicity. However, metal oxides or hydroxides were formed along with $C_6H_{11}NH_3^+NO_3^-$, which reflects the Brønsted-Lowry basicity of CHA ($pK_b = 3.36$, Solomons, 1996). This base strength makes CHA suitable for the preparation of several salts of anions and complex anions through the formation of the primary ammonium cation ($C_6H_{11}NH_3^+$) (Jones *et al.*, 1998; Kolev *et al.*, 2007; Lock *et al.*, 1981; Muthamizhchelvan *et al.*, 2005; Shimada *et al.*, 1955; Smith *et al.*, 1994; Wang *et al.*, 2005; Yun *et al.*, 2004). This Brønsted-Lowry behavior was responsible for the formation of the present compound, (I) (Fig. 1), which is dangerous to prepare from a direct reaction between CHA and nitric acid (HNO_3) because CHA reacts violently with strong acids or oxidizing agents and may cause fire and explosion (Chang, 2008; Patnaik, 2007).

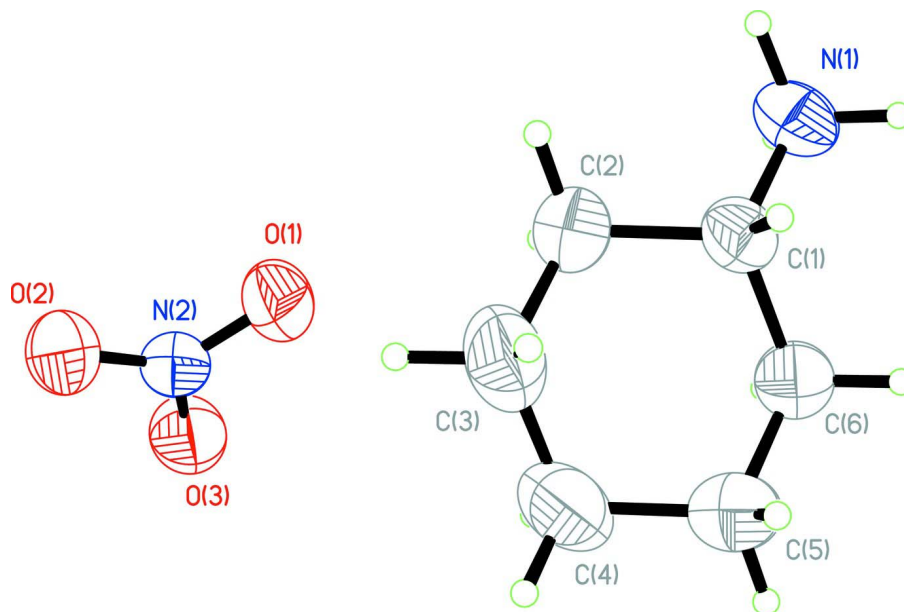
The asymmetric unit of the title compound contains one cyclohexylammonium cation (C1—C6/N1) and one nitrate anion (N2/O1—O3). The cyclohexane ring adopts a chair conformation, with puckering parameters: $Q = 0.5668(17)$ Å, $\theta = 179.29(17)^\circ$, and $\varphi = 276(21)^\circ$ (Cremer & Pople, 1975). The ammonium functional group is at an equatorial position to minimize 1,3 and 1,5 di-axial interactions. The bond lengths (Allen *et al.*, 1987) and bond angles are in the normal ranges and are comparable with those reported earlier for similar compounds (Shimada *et al.*, 1955; Smith *et al.*, 1994; Odendal *et al.*, 2010). Each proton of the ammonium group is hydrogen-bonded to two oxygen atoms of the nitrate ion. These intermolecular N—H \cdots O hydrogen bonds (Table 2) generate a three-dimensional network (Fig. 2).

S2. Experimental

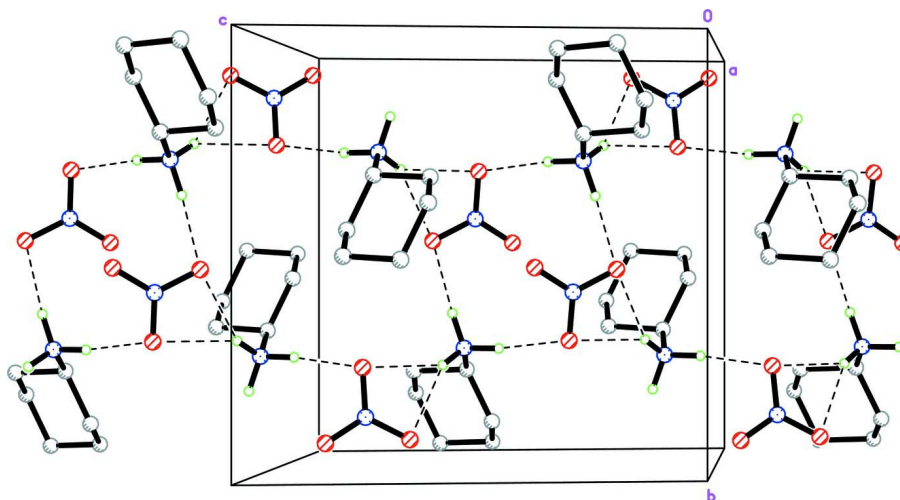
The title compound $C_6H_{11}NH_3^+NO_3^-$ was obtained as a by-product upon combining 60 ml, 0.5 M of metal nitrate (metal = Mg^{2+} , Al^{3+} , Cr^{3+} , Mn^{2+} , Fe^{3+} , Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} , or Cd^{2+}) with 20 ml, 3.0 M (for divalent metal) or 4.5 M (for trivalent metal) CHA in aqueous or ethanolic media. Depending on the identity of M, a metal hydroxide or oxide was precipitated. Filtering this precipitate resulted in a clear filtrate, which upon the gradual evaporation of the solvent at room temperature resulted in the deposition of beautiful, colorless crystals of $HCHA^+NO_3^-$. The chemical composition of these crystals was determined by C, H, N elemental microanalysis: (%C: 44.47 exp; 44.43 cal.), (%H: 8.70 exp.; 8.72 cal.), (%N: 17.26 exp.; 17.28 cal.), and (%O: 29.61 exp.; 29.59 cal.).

S3. Refinement

The nitrogen-bound H-atoms were located in a difference Fourier map and were fixed at their found positions (N—H = 0.8498, 0.9440 and 0.9724 Å), with $U_{iso}(H) = 1.2 U_{eq}(N)$. Other H atoms were positioned geometrically (C—H 0.97–0.98 Å) and refined using a riding model with $U_{iso}(H) = 1.2 U_{eq}(C)$


Figure 1

Molecular structure of the compound, with atom labels and 50% probability displacement ellipsoids for the non-H atoms.


Figure 2

Crystal packing of the title compound, showing the hydrogen bonding interactions as dashed lines.

Cyclohexylammonium nitrate

Crystal data

$C_6H_{14}N^+ \cdot NO_3^-$

$M_r = 162.19$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/bc$

$a = 8.9322(9) \text{ \AA}$

$b = 9.9010(9) \text{ \AA}$

$c = 10.3951(10) \text{ \AA}$

$\beta = 103.866(2)^\circ$

$V = 892.53(15) \text{ \AA}^3$

$Z = 4$

$F(000) = 352$

$D_x = 1.207 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 11857 reflections

$\theta = 2.4\text{--}28.3^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 294$ K $0.39 \times 0.15 \times 0.14$ mm
 Block, colorless

Data collection

Bruker APEXII CCD diffractometer Radiation source: fine-focus sealed tube Graphite monochromator φ and ω scans Absorption correction: multi-scan (SADABS; Sheldrick, 1996) $T_{\min} = 0.964$, $T_{\max} = 0.987$	2214 measured reflections 2214 independent reflections 1750 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.000$ $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.4^\circ$ $h = -11 \rightarrow 11$ $k = 0 \rightarrow 13$ $l = 0 \rightarrow 13$
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Refinement

Refinement on F^2 Least-squares matrix: full $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.121$ $S = 1.09$ 2214 reflections 101 parameters 0 restraints 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 $w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.0786P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.005$ $\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$ Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$ Extinction coefficient: 0.042 (6)
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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.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.94737 (12)	0.24027 (11)	0.84734 (10)	0.0591 (3)
H1	1.0040	0.2396	0.9397	0.071*
H2	0.9334	0.1504	0.8165	0.071*
H3	1.0001	0.2818	0.8017	0.071*
C1	0.79115 (13)	0.30167 (11)	0.83331 (11)	0.0504 (3)
H4	0.7312	0.2428	0.8779	0.060*
C2	0.80481 (15)	0.43882 (13)	0.89905 (13)	0.0619 (3)
H5	0.8701	0.4967	0.8605	0.074*
H6	0.8523	0.4293	0.9928	0.074*
C3	0.64691 (18)	0.50274 (15)	0.88097 (18)	0.0806 (4)
H7	0.6580	0.5927	0.9190	0.097*
H8	0.5855	0.4495	0.9276	0.097*

C4	0.56468 (19)	0.51139 (15)	0.7354 (2)	0.0887 (5)
H9	0.4622	0.5482	0.7270	0.106*
H10	0.6208	0.5721	0.6906	0.106*
C5	0.55222 (17)	0.37454 (16)	0.67001 (17)	0.0833 (5)
H11	0.5052	0.3842	0.5762	0.100*
H12	0.4864	0.3168	0.7081	0.100*
C6	0.70975 (16)	0.30923 (13)	0.68820 (12)	0.0638 (3)
H13	0.6979	0.2189	0.6509	0.077*
H14	0.7716	0.3614	0.6412	0.077*
O1	0.90382 (12)	0.78324 (9)	0.87802 (9)	0.0694 (3)
O2	0.82965 (10)	0.96820 (10)	0.95391 (9)	0.0681 (3)
O3	0.87733 (12)	0.96351 (10)	0.75996 (9)	0.0729 (3)
N2	0.86827 (10)	0.90604 (10)	0.86507 (9)	0.0529 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0641 (6)	0.0561 (5)	0.0586 (6)	0.0105 (4)	0.0177 (4)	0.0154 (4)
C1	0.0543 (6)	0.0449 (5)	0.0521 (6)	0.0010 (4)	0.0129 (5)	0.0065 (4)
C2	0.0651 (7)	0.0546 (7)	0.0635 (7)	-0.0030 (5)	0.0108 (6)	-0.0057 (5)
C3	0.0777 (9)	0.0615 (8)	0.1048 (12)	0.0089 (7)	0.0265 (9)	-0.0157 (8)
C4	0.0674 (8)	0.0606 (8)	0.1274 (15)	0.0139 (7)	0.0022 (9)	0.0051 (8)
C5	0.0720 (8)	0.0705 (9)	0.0906 (10)	0.0057 (7)	-0.0135 (7)	0.0017 (8)
C6	0.0754 (8)	0.0569 (7)	0.0535 (7)	0.0052 (6)	0.0041 (6)	0.0008 (5)
O1	0.0868 (6)	0.0492 (5)	0.0696 (6)	0.0075 (4)	0.0139 (5)	0.0001 (4)
O2	0.0688 (5)	0.0736 (6)	0.0650 (5)	0.0105 (4)	0.0221 (4)	-0.0094 (4)
O3	0.0975 (7)	0.0663 (6)	0.0549 (5)	0.0129 (5)	0.0181 (5)	0.0090 (4)
N2	0.0479 (5)	0.0538 (5)	0.0536 (5)	0.0040 (4)	0.0051 (4)	-0.0018 (4)

Geometric parameters (Å, °)

N1—C1	1.4968 (15)	C3—H8	0.9700
N1—H1	0.9724	C4—C5	1.508 (2)
N1—H2	0.9440	C4—H9	0.9700
N1—H3	0.8498	C4—H10	0.9700
C1—C6	1.5112 (16)	C5—C6	1.519 (2)
C1—C2	1.5119 (17)	C5—H11	0.9700
C1—H4	0.9800	C5—H12	0.9700
C2—C3	1.5159 (19)	C6—H13	0.9700
C2—H5	0.9700	C6—H14	0.9700
C2—H6	0.9700	O1—N2	1.2556 (13)
C3—C4	1.518 (3)	O2—N2	1.2261 (12)
C3—H7	0.9700	O3—N2	1.2516 (13)
C1—N1—H1	110.6	H7—C3—H8	108.0
C1—N1—H2	107.8	C5—C4—C3	111.37 (13)
H1—N1—H2	108.9	C5—C4—H9	109.4
C1—N1—H3	112.2	C3—C4—H9	109.4

H1—N1—H3	109.1	C5—C4—H10	109.4
H2—N1—H3	108.2	C3—C4—H10	109.4
N1—C1—C6	109.37 (10)	H9—C4—H10	108.0
N1—C1—C2	110.38 (10)	C4—C5—C6	111.12 (12)
C6—C1—C2	111.97 (10)	C4—C5—H11	109.4
N1—C1—H4	108.3	C6—C5—H11	109.4
C6—C1—H4	108.3	C4—C5—H12	109.4
C2—C1—H4	108.3	C6—C5—H12	109.4
C1—C2—C3	110.31 (11)	H11—C5—H12	108.0
C1—C2—H5	109.6	C1—C6—C5	110.81 (12)
C3—C2—H5	109.6	C1—C6—H13	109.5
C1—C2—H6	109.6	C5—C6—H13	109.5
C3—C2—H6	109.6	C1—C6—H14	109.5
H5—C2—H6	108.1	C5—C6—H14	109.5
C2—C3—C4	111.14 (13)	H13—C6—H14	108.1
C2—C3—H7	109.4	O2—N2—O3	121.20 (10)
C4—C3—H7	109.4	O2—N2—O1	120.99 (10)
C2—C3—H8	109.4	O3—N2—O1	117.79 (10)
C4—C3—H8	109.4		
N1—C1—C2—C3	178.21 (11)	C3—C4—C5—C6	-55.5 (2)
C6—C1—C2—C3	56.11 (15)	N1—C1—C6—C5	-178.51 (11)
C1—C2—C3—C4	-55.73 (16)	C2—C1—C6—C5	-55.83 (15)
C2—C3—C4—C5	56.08 (19)	C4—C5—C6—C1	55.08 (18)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1 ⁱ	0.97	1.89	2.8553 (14)	172
N1—H2...O3 ⁱⁱ	0.94	1.97	2.9074 (15)	172
N1—H3...O1 ⁱⁱⁱ	0.85	2.24	2.9880 (15)	148
N1—H3...O3 ⁱⁱⁱ	0.85	2.28	3.0689 (15)	155

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $x, y-1, z$; (iii) $-x+2, y-1/2, -z+3/2$.