

N,N',N''-Tricyclohexylguanidinium chloride

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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.067
 wR factor = 0.162
Data-to-parameter ratio = 17.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

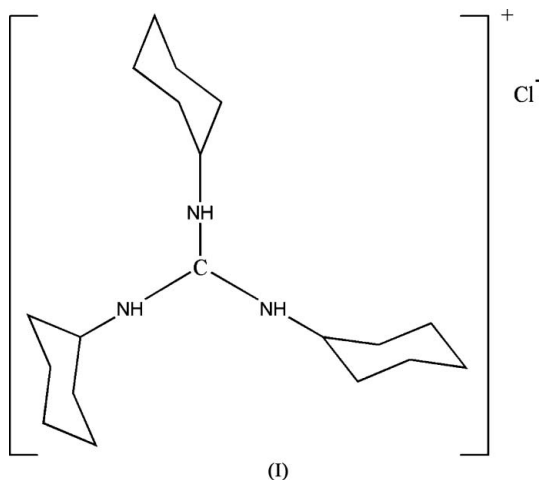
In the title crystal structure, $\text{C}_{19}\text{H}_{36}\text{N}_3^+\cdot\text{Cl}^-$, the central C atom of the *N,N',N''*-tricyclohexylguanidinium ion and the chloride ion both lie on positions of site symmetry 3. Weak $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds [$\text{N}\cdots\text{Cl} = 3.539$ (3) Å] link anions and cations, forming a three-dimensional network.

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Comment

Guanidines are strong bases that find applications in a large number of organic reactions widely employed in organic synthesis, including carbon-carbon bond formation and transesterification of vegetable oils (Horvath, 1996; Gobbi & Frenking, 1993; Vargas *et al.*, 1998). Coupled with the increasing emphasis on the development of environmentally friendly catalysts, the heterogenization of these bases is a desirable goal (Tanatani *et al.*, 1998; Sercheli *et al.*, 1999). Here we report the the crystal structure of the title compound, (I).



The title compound comprises an *N,N',N''*-tricyclohexylguanidinium cation and a chloride anion (Fig. 1). C1 and Cl1 both lie on positions of site symmetry 3. In the *N,N',N''*-tricyclohexylguanidinium cation, the sum of the angles at atom C1 confirms the sp^2 hybridization of this atom. The unique cyclohexyl group is in a chair conformation. In the crystal structure, anions and cations are linked by intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming a three-dimensional network (Table 2).

Experimental

A DMF solution (10 ml) of *N,N',N''*-tricyclohexylguanidine (0.1 mmol, 0.31 g) was added dropwise to a stirred aqueous solution (10 ml) of hydrochloric acid (0.2 mmol, 0.07 g) at 253 K. The reaction

mixture was filtered and the filtrate allowed to stand for approximately two weeks until colorless single crystals formed.

Crystal data

$C_{19}H_{36}N_3^+ \cdot Cl^-$
 $M_r = 341.96$
 Cubic, $P2_13$
 $a = 12.6411 (5) \text{ \AA}$
 $V = 2020.02 (14) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.124 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation

Cell parameters from 2136 reflections
 $\theta = 2.5\text{--}24.1^\circ$
 $\mu = 0.19 \text{ mm}^{-1}$
 $T = 298 (2) \text{ K}$
 Block, colorless
 $0.34 \times 0.28 \times 0.12 \text{ mm}$

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.931$, $T_{\max} = 0.972$
 10792 measured reflections

1221 independent reflections
 1146 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\text{max}} = 25.2^\circ$
 $h = -15 \rightarrow 11$
 $k = -15 \rightarrow 14$
 $l = -13 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.162$
 $S = 1.29$
 1221 reflections
 70 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.4253P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983), 524 Friedel pairs
 Flack parameter: 0.16 (2)

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—C1	1.331 (3)	C3—C4	1.530 (6)
N1—C2	1.460 (4)	C4—C5	1.475 (8)
C2—C3	1.500 (6)	C5—C6	1.502 (8)
C2—C7	1.504 (6)	C6—C7	1.545 (8)
C1—N1—C2	125.6 (3)	N1—C2—C7	110.3 (3)
N1—C2—C3	110.7 (3)	C3—C2—C7	110.0 (3)
C7—C2—C3—C4	58.9 (5)	C4—C5—C6—C7	−55.7 (7)
C2—C3—C4—C5	−59.5 (5)	C3—C2—C7—C6	−57.9 (6)
C3—C4—C5—C6	57.9 (6)	C5—C6—C7—C2	56.0 (7)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots Cl1	0.86	2.72	3.539 (3)	160

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $Csp^3-H = 0.93 \text{ \AA}$ with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ and $N-H = 0.86 \text{ \AA}$ with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N)$.

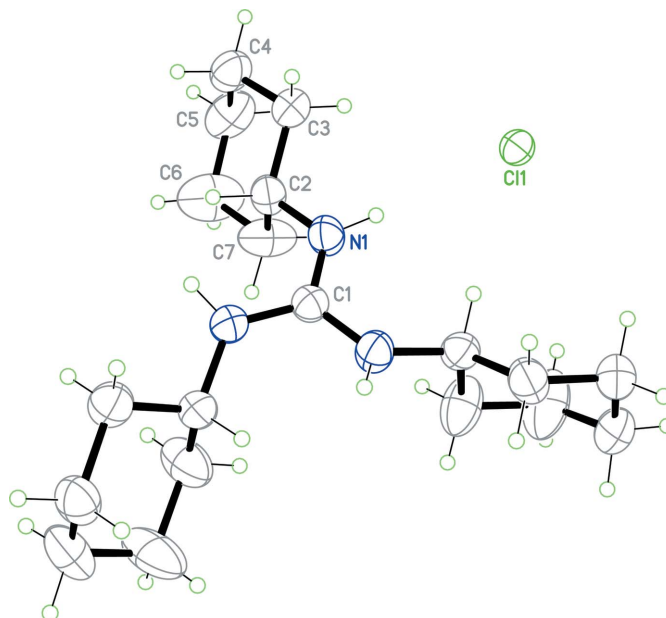


Figure 1

The structure of (I) with displacement ellipsoids drawn at the 30% probability level and H atoms shown as spheres of arbitrary radii. In the cation, only the symmetry-unique atoms are labelled, the others being related by three-fold rotation ($\frac{2}{3} - z, 2 - x, y - \frac{1}{2}$ and $2 - y, \frac{1}{2} + z, \frac{2}{3} - x$).

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXL97.

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