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3-Chloroquinuclidinium chloride

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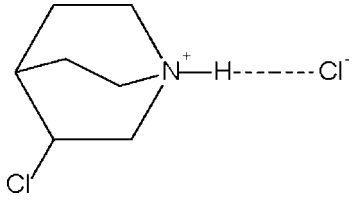
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(N–C) = 0.004 Å; disorder in main residue; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 13.0.

The cation of the title compound, $C_7H_{13}ClN^+\cdot Cl^-$, forms a linear hydrogen bond to the chloride anion. The cation is disordered about a mirror plane.

Related literature

For isomeric 4-chloroquinuclidinium chloride, see: Kurahashi *et al.* (1980), which also reports the parent quinuclidinium chloride.



Experimental

Crystal data

$C_7H_{13}ClN^+\cdot Cl^-$
M_r = 182.10
Orthorhombic, *Pnma*
a = 9.379 (1) Å
b = 8.067 (1) Å
c = 11.482 (2) Å
V = 868.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.68 mm^{-1}
T = 100 (2) K
0.15 × 0.08 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.872, T_{max} = 1.000
(expected range = 0.855–0.980)
5307 measured reflections
1068 independent reflections
856 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.113
S = 1.02
1068 reflections
82 parameters
58 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.88 (1)	2.13 (1)	3.008 (3)	175 (4)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2068).

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supplementary materials

Acta Cryst. (2008). E**64**, o911 [**doi:10.1107/S1600536808011434**]

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Comment

4-Chloroquinuclidinium chloride features an N–H···Cl hydrogen bond between the cation and anion. The N–C and C–C bonds are somewhat shorter than those in the unsubstituted salt, and this has been attributed to the electron-withdrawing effect of the chlorine substituent (Kurahashi *et al.*, 1980). The present isomeric compound (Scheme I) is expected to show this feature; however, owing to disorder, the effect cannot be unambiguously observed even at low temperature. The cation forms a linear hydrogen bond [N–H···Cl 3.008 (3) Å] to the chloride; the cation is disordered about a mirror plane (Fig. 1).

Experimental

The commercially available compound was a crystalline. A large block was cut into a smaller specimen.

Refinement

The cation is disordered about a mirror plane in the carbon atoms except C1 atom. The N1 and C1 atoms, which lie on this symmetry element, were refined with their normal half occupancies. The other carbon atoms were refined with half occupancies, subject to N–C being restrained to 1.49±0.01 Å and C–C to 1.54±0.01 Å. Additionally, the 1,3-related distances were restrained from 2.43±0.01 Å, to 2.47±0.01 Å as well as 2.52±-0.01 Å. The anisotropic temperature factors of the disordered carbon were restrained to be nearly isotropic but the N–H distance was restrained to 0.88±0.01 Å.

Carbon-bound H-atoms were placed in calculated positions (C—H 0.99 to 1.00 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C). The ammonium H-atom was located in a difference Fourier map, and was refined with an N–H distance restraint of 0.88±0.01 Å; its temperature factor was freely refined.

Figures

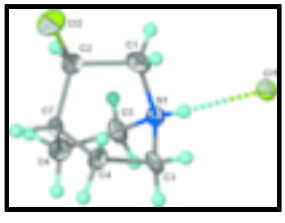

Fig. 1. Thermal ellipsoid plot of the two independent molecules of 2-chloroquinuclidinium chloride at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The dashed lines denote the hydrogen bond.

3-Chloroquinuclidinium chloride

Crystal data

$C_7H_{13}ClN^+\cdot Cl^-$ $F_{000} = 392$

M_r = 182.10	D_x = 1.408 Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 909 reflections
a = 9.379 (1) Å	θ = 3.1–22.9º
b = 8.067 (1) Å	μ = 0.68 mm^{-1}
c = 11.482 (2) Å	*T* = 100 (2) K
V = 868.7 (2) Å^3	Block, colorless
Z = 4	0.15 × 0.08 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer	1068 independent reflections
Radiation source: fine-focus sealed tube	856 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 100(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −12→8
T_{min} = 0.872, T_{max} = 1.000	*k* = −10→10
5307 measured reflections	*l* = −14→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.113	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.8408P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
1068 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
82 parameters	$\Delta\rho_{min}$ = −0.57 e Å^{-3}
58 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.69230 (8)	0.2500	0.57828 (7)	0.0218 (2)	
Cl2	0.10483 (11)	0.2500	0.30411 (8)	0.0374 (3)	
N1	0.3718 (3)	0.2500	0.5682 (2)	0.0190 (6)	
H1	0.4648 (12)	0.2500	0.576 (3)	0.035 (12)*	
C1	0.3382 (3)	0.2500	0.4416 (3)	0.0331 (9)	
H1A	0.4092	0.1832	0.3983	0.040*	0.50

H1B	0.3397	0.3645	0.4106	0.040*	0.50
C2	0.1846 (4)	0.1722 (5)	0.4282 (3)	0.0184 (9)	0.50
H2	0.1947	0.0495	0.4194	0.022*	
C3	0.2957 (7)	0.3924 (13)	0.6253 (9)	0.0204 (17)	0.50
H3A	0.3397	0.4988	0.6019	0.024*	0.50
H3B	0.3013	0.3825	0.7111	0.024*	0.50
C4	0.1402 (5)	0.3855 (6)	0.5851 (5)	0.0212 (11)	0.50
H4A	0.1248	0.4653	0.5209	0.025*	0.50
H4B	0.0763	0.4159	0.6503	0.025*	0.50
C5	0.3285 (7)	0.0917 (13)	0.6251 (10)	0.024 (2)	0.50
H5A	0.3698	0.0857	0.7043	0.029*	0.50
H5B	0.3651	−0.0034	0.5795	0.029*	0.50
C6	0.1650 (5)	0.0823 (7)	0.6325 (4)	0.0244 (12)	0.50
H6A	0.1323	0.1111	0.7120	0.029*	0.50
H6B	0.1316	−0.0310	0.6139	0.029*	0.50
C7	0.1058 (4)	0.2079 (5)	0.5430 (4)	0.0205 (12)	0.50
H7	0.0007	0.1931	0.5330	0.025*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0157 (4)	0.0284 (4)	0.0212 (4)	0.000	−0.0006 (3)	0.000
Cl2	0.0340 (5)	0.0546 (6)	0.0236 (5)	0.000	−0.0111 (4)	0.000
N1	0.0145 (13)	0.0241 (13)	0.0184 (14)	0.000	−0.0020 (10)	0.000
C1	0.0176 (17)	0.064 (3)	0.0182 (19)	0.000	0.0006 (13)	0.000
C2	0.019 (2)	0.0171 (19)	0.019 (2)	0.0018 (17)	−0.0032 (16)	0.0013 (17)
C3	0.019 (3)	0.017 (3)	0.025 (3)	−0.004 (3)	0.008 (3)	0.004 (2)
C4	0.015 (2)	0.022 (3)	0.026 (3)	0.004 (2)	−0.004 (2)	−0.006 (2)
C5	0.017 (3)	0.019 (3)	0.036 (4)	0.000 (3)	0.007 (3)	−0.001 (3)
C6	0.028 (3)	0.026 (3)	0.019 (3)	−0.003 (2)	−0.002 (2)	0.004 (2)
C7	0.0108 (17)	0.030 (4)	0.021 (2)	−0.0037 (16)	0.0014 (15)	−0.0070 (18)

Geometric parameters (Å, °)

Cl2—C2	1.727 (4)	C3—H3B	0.9900
N1—C5	1.491 (7)	C4—C7	1.546 (6)
N1—C1	1.488 (4)	C4—H4A	0.9900
N1—C3	1.502 (7)	C4—H4B	0.9900
N1—H1	0.88 (1)	C5—C6	1.538 (7)
C1—C2	1.579 (4)	C5—H5A	0.9900
C1—H1A	0.9900	C5—H5B	0.9900
C1—H1B	0.9900	C6—C7	1.546 (5)
C2—C7	1.539 (5)	C6—H6A	0.9900
C2—H2	1.0000	C6—H6B	0.9900
C3—C4	1.531 (7)	C7—H7	1.0000
C3—H3A	0.9900		
C5—N1—C1	111.7 (5)	C3—C4—C7	109.1 (4)
C5—N1—C3	109.5 (3)	C3—C4—H4A	109.9

C1—N1—C3	109.0 (4)	C7—C4—H4A	109.9
C5—N1—H1	103.1 (13)	C3—C4—H4B	109.9
C1—N1—H1	108 (3)	C7—C4—H4B	109.9
C3—N1—H1	115.3 (14)	H4A—C4—H4B	108.3
N1—C1—C2	106.8 (2)	N1—C5—C6	109.8 (5)
N1—C1—H1A	110.4	N1—C5—H5A	109.7
C2—C1—H1A	110.4	C6—C5—H5A	109.7
N1—C1—H1B	110.4	N1—C5—H5B	109.7
C2—C1—H1B	110.4	C6—C5—H5B	109.7
H1A—C1—H1B	108.6	H5A—C5—H5B	108.2
C7—C2—C1	106.3 (3)	C5—C6—C7	106.8 (4)
C7—C2—Cl2	115.5 (3)	C5—C6—H6A	110.4
C1—C2—Cl2	109.4 (2)	C7—C6—H6A	110.4
C7—C2—H2	108.5	C5—C6—H6B	110.4
C1—C2—H2	108.5	C7—C6—H6B	110.4
Cl2—C2—H2	108.5	H6A—C6—H6B	108.6
N1—C3—C4	107.1 (4)	C2—C7—C4	109.9 (3)
N1—C3—H3A	110.3	C2—C7—C6	106.0 (3)
C4—C3—H3A	110.3	C4—C7—C6	108.9 (3)
N1—C3—H3B	110.3	C2—C7—H7	110.6
C4—C3—H3B	110.3	C4—C7—H7	110.6
H3A—C3—H3B	108.6	C6—C7—H7	110.6
C5—N1—C1—C2	42.7 (4)	N1—C5—C6—C7	18.9 (10)
C3—N1—C1—C2	−78.5 (4)	C1—C2—C7—C4	40.7 (4)
N1—C1—C2—C7	27.3 (3)	Cl2—C2—C7—C4	−80.8 (4)
N1—C1—C2—Cl2	152.63 (17)	C1—C2—C7—C6	−76.9 (4)
C5—N1—C3—C4	−73.4 (6)	Cl2—C2—C7—C6	161.6 (3)
C1—N1—C3—C4	49.1 (8)	C3—C4—C7—C2	−70.0 (6)
N1—C3—C4—C7	21.4 (9)	C3—C4—C7—C6	45.7 (6)
C1—N1—C5—C6	−71.2 (9)	C5—C6—C7—C2	49.6 (7)
C3—N1—C5—C6	49.7 (7)	C5—C6—C7—C4	−68.6 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1	0.88 (1)	2.13 (1)	3.008 (3)	175 (4)

Fig. 1