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Redetermination of *trans*-cyclohexane-1,4-diammonium dichloride

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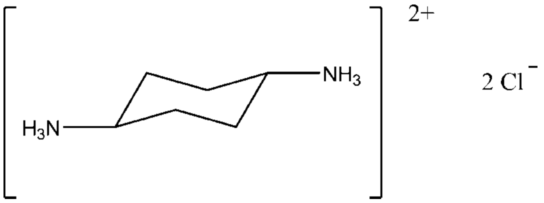
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.072; data-to-parameter ratio = 22.4.

A redetermination of the crystal structure of the title compound, $C_6H_{16}N_2^{2+}\cdot 2Cl^-$, was undertaken. All atomic coordinates including those of the H atoms were refined freely. The cation is located on a centre of symmetry. Important for the crystal structure are wavy hydrogen-bonded layers that are formed by ammonium groups and chloride anions, giving hydrogen-bonded $R_6^3(12)$ rings.

Related literature

For previous structure determinations, see: Dunitz & Strickler (1965, 1966). For the isostructural cyclohexane-1,4-diammonium dibromide, see: Rademeyer (2006). For hydrogen-bond motifs, see: Etter *et al.* (1990); Rademeyer (2006).



Experimental

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2Cl^-$	V = 490.39 (16) Å^3
M_r = 187.11	Z = 2
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 5.2550 (11) Å	μ = 0.60 mm^{-1}
b = 14.890 (3) Å	T = 293 (2) K
c = 6.3604 (12) Å	0.30 × 0.24 × 0.20 mm
β = 99.824 (18)°	

Data collection

Stoe STADI CCD diffractometer	1766 independent reflections
Absorption correction: none	1562 reflections with $I > 2\sigma(I)$
13502 measured reflections	$R_{\rm int}$ = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038	79 parameters
$wR(F^2)$ = 0.072	All H-atom parameters refined
S = 1.03	$\Delta\rho_{\rm max}$ = 0.41 e Å^{-3}
1766 reflections	$\Delta\rho_{\rm min}$ = −0.28 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H3⋯Cl1$^{\rm ii}$	0.88 (2)	2.30 (2)	3.1734 (15)	170.3 (16)
N1—H2⋯Cl1$^{\rm iii}$	0.86 (2)	2.33 (2)	3.1833 (13)	171.9 (17)
N1—H1⋯Cl1	0.93 (2)	2.23 (2)	3.1584 (13)	173.9 (16)

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

Data collection: *CrysAlis CCD* (Kuma Diffraction, 2000); cell refinement: *CrysAlis RED* (Kuma Diffraction, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GD2030).

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supporting information

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Redetermination of *trans*-cyclohexane-1,4-diammonium dichloride

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S1. Comment

The title compound was first crystallographically characterized by Dunitz and coworkers in 1966 (Dunitz & Strickler,1965, 1966). This quality structure determination only lacks the fact that all hydrogen atom positions, especially those of the ammonium group, were introduced into the structure model on the basic of geometrically calculated positions, with the N—H and the C—H distances set to 1.1 Å. For X-ray data refinement of hydrogen atom positions significantly shorter values are commonly found. We now describe an improved structure model - the hydrogen atoms were reliably found and refined from quality X-ray data.

A standard refinement using reflections up to 50 ° / 2Θ gave the following values: R^2 = 6.01, R_1 = 2.97, GooF = 1.197. Using data with reflections up to 65°/2Θ a more stable refinement is possible and the standard uncertainies of the N—H-distances are smaller.

The title structure consists of hydrogen bonded hydrophilic layers in the *ac*-plane. These wavy layers are built by an annulated ring-motif ($R^3_6(12)$; Etter, 1990) constructed by three chloride anions and three ammonium groups (Fig. 3). Each ammonium group donates three hydrogen bonds of only slightly different strength to neighbouring chloride anions (Tab. 2, Fig. 1 + 2). The title compound is therefore isostructual but not isotypic to the cyclohexane-1,4-diammonium dibromide (Rademeyer, 2006).

In terms of crystal engineering the structure of the title compound is dominated by the hydrogen bonded layers. The aliphatic cyclohexane-1,4-diyl fragments connect these layers. According to the positions of the ammonium groups in the hydrogen bonded network the cyclohexyl-fragments do not appear cloesly packed (Fig. 3).

S2. Experimental

trans-Cyclohexane-1,4-diammonium dichloride was prepared by the reaction of 1,4-diaminocyclohexane (+99%, Aldrich, 0.11 g) and hydrochloric acid (37%) at room temperature. From this colourless solution small block shaped crystals were obtained.

S3. Refinement

All hydrogen atom positions were obtained from difference fourier maps, all hydrogen atoms were refined freely and with an individual isotropic displacement parameter for each (H—*X* distance range: 0.88–1.01 A).

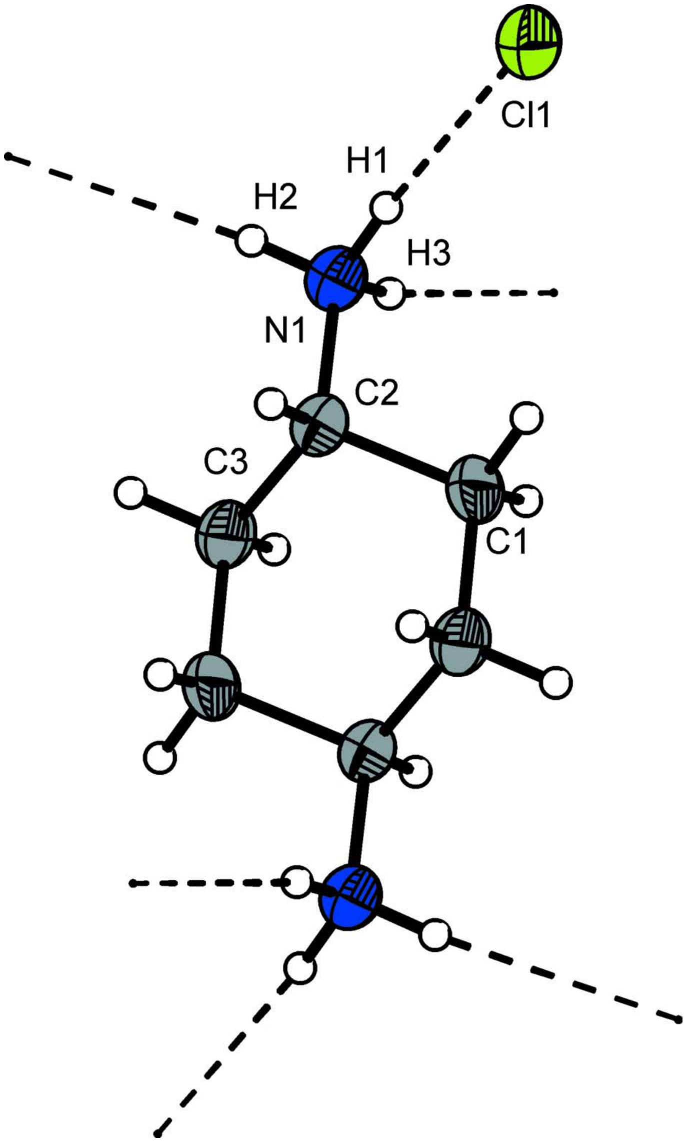

Figure 1

The structure of the title compound (displacement ellipsoids at the 40% probability level, H-atoms drawn with arbitrary radius).Thin dashed lines show hydrogen bonds to neighbouring chloride anions. The atoms of the asymmetric unit are labeled.

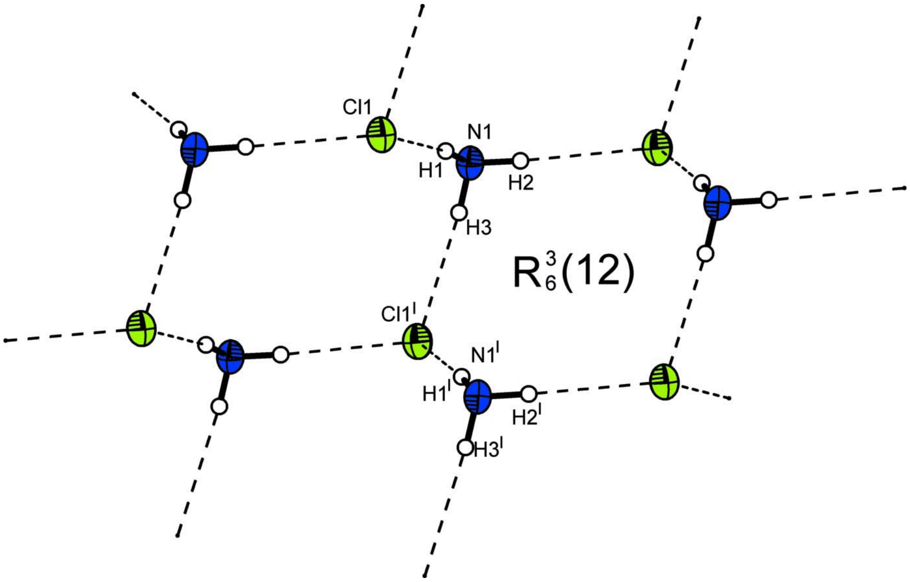

Figure 2

Hydrogen bonded layer in the *ac*-plane of the title structure, constructed from annulated R^3_6(12) motifs. (Symmetry code ′ = -1/2 + *x*, 0,5 - *y*, -1/2 + *z*)

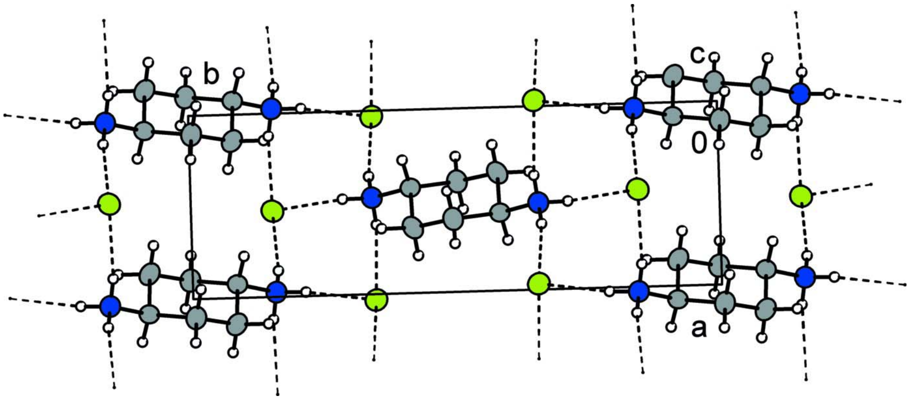

Figure 3

Crystal packing seen along the c direction. Hydrogen bonding interactions are shown as dotted lines.

trans-cyclohexane-1,4-diammonium dichloride

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2Cl^-$
M_r = 187.11
Monoclinic, $P2_1/n$
a = 5.2550 (11) Å
b = 14.890 (3) Å
c = 6.3604 (12) Å
β = 99.824 (18)°
V = 490.39 (16) Å^3
Z = 2

$F(000)$ = 200
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1610 reflections
θ = 4.8–17.4°
μ = 0.60 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.24 × 0.20 mm

Data collection

Stoe STADI CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
13502 measured reflections
1766 independent reflections
1562 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\rm max} = 32.5°$, $\theta_{\rm min} = 4.3°$
$h = -7 \rightarrow 7$
$k = -22 \rightarrow 22$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.072$
$S = 1.04$
1766 reflections
79 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + 0.3P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.41$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.174 (6)

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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Cl1	0.46825 (6)	0.15413 (2)	0.46296 (5)	0.03915 (12)
N1	0.5260 (2)	0.34260 (8)	0.24820 (19)	0.0338 (2)
H1	0.522 (3)	0.2870 (13)	0.316 (3)	0.051 (5)*
H2	0.642 (4)	0.3377 (12)	0.168 (3)	0.057 (5)*
H3	0.377 (4)	0.3498 (12)	0.163 (3)	0.050 (5)*
C1	0.6023 (3)	0.50463 (8)	0.3001 (2)	0.0335 (3)
H11	0.448 (3)	0.5150 (11)	0.198 (3)	0.041 (4)*
H12	0.738 (4)	0.5022 (11)	0.221 (3)	0.043 (4)*
C2	0.5751 (2)	0.41590 (8)	0.41008 (18)	0.0281 (2)
H21	0.726 (3)	0.4018 (10)	0.503 (2)	0.029 (3)*
C3	0.3565 (3)	0.41864 (9)	0.5386 (2)	0.0358 (3)
H31	0.203 (3)	0.4276 (11)	0.445 (3)	0.042 (4)*
H32	0.355 (3)	0.3590 (11)	0.613 (3)	0.044 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.03850 (18)	0.03987 (18)	0.04168 (19)	−0.00250 (12)	0.01427 (13)	0.00190 (13)
N1	0.0361 (5)	0.0330 (5)	0.0350 (5)	−0.0006 (4)	0.0137 (4)	−0.0018 (4)
C1	0.0416 (6)	0.0331 (6)	0.0301 (6)	−0.0022 (5)	0.0179 (5)	0.0026 (4)
C2	0.0275 (5)	0.0315 (5)	0.0267 (5)	0.0006 (4)	0.0082 (4)	0.0021 (4)
C3	0.0394 (6)	0.0341 (6)	0.0390 (6)	−0.0078 (5)	0.0211 (5)	−0.0008 (5)

Geometric parameters (Å, º)

N1—C2	1.4924 (16)	C2—C3	1.5201 (16)
C1—C2	1.5130 (17)	C2—H21	0.928 (15)
C1—C3^{i}	1.5259 (18)	C3—C1^{i}	1.5259 (18)
C1—H11	0.959 (17)	C3—H31	0.927 (17)
C1—H12	0.941 (19)	C3—H32	1.008 (17)
C2—N1—H1	110.2 (11)	N1—C2—C3	109.47 (10)
C2—N1—H2	114.4 (13)	N1—C2—C1	109.79 (10)
H1—N1—H2	105.8 (16)	C3—C2—C1	111.39 (10)
C2—N1—H3	111.6 (12)	N1—C2—H21	107.7 (9)
H1—N1—H3	107.8 (16)	C3—C2—H21	107.6 (9)
H2—N1—H3	106.8 (18)	C1—C2—H21	110.8 (9)
C2—C1—C3^{i}	110.91 (10)	C2—C3—C1^{i}	110.36 (10)
C2—C1—H11	108.6 (10)	C2—C3—H31	108.1 (11)
C3^{i}—C1—H11	110.0 (10)	C1^{i}—C3—H31	109.5 (10)
C2—C1—H12	110.8 (10)	C2—C3—H32	107.4 (10)
C3^{i}—C1—H12	110.9 (10)	C1^{i}—C3—H32	110.8 (10)
H11—C1—H12	105.6 (15)	H31—C3—H32	110.6 (15)
C3^{i}—C1—C2—N1	178.07 (10)	N1—C2—C3—C1^{i}	−177.94 (11)
C3^{i}—C1—C2—C3	56.65 (16)	C1—C2—C3—C1^{i}	−56.34 (16)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H3···Cl1^{ii}	0.88 (2)	2.30 (2)	3.1734 (15)	170.3 (16)
N1—H2···Cl1^{iii}	0.86 (2)	2.33 (2)	3.1833 (13)	171.9 (17)
N1—H1···Cl1	0.93 (2)	2.23 (2)	3.1584 (13)	173.9 (16)

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