

Ethylenediammonium dichloriodide chloride

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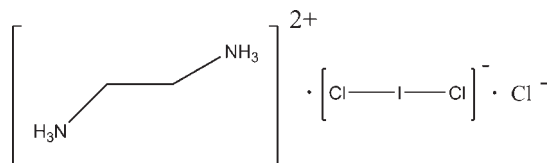
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.023; wR factor = 0.056; data-to-parameter ratio = 27.2.

The asymmetric unit of the crystal structure of the title compound, $\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot \text{Cl}_2\text{I}^- \cdot \text{Cl}^-$, contains two ethylenediammonium cations, two $[\text{ICl}_2]^-$ anions and two Cl^- anions, of which one cation, one $[\text{ICl}_2]^-$ anion and one Cl^- anion have site symmetry 2, with the mid-point of the C—C bond of the cation, the I atom of $[\text{ICl}_2]^-$ anion and the Cl^- anion located on the twofold rotation axes. The two independent cations show different conformations, the N—C—N torsion angles being 160.1 (2) and -73.1 (4)°. The crystal structure is stabilized by extensive intermolecular N—H...Cl hydrogen bonding.

Related literature

For general background to combining protonated aromatic nitrogen bases with halide or polyhalide ions, see: Tucker & Kroon (1973); Bandoli *et al.* (1978). For Cl—I bond lengths and Cl—I—Cl bond angles, see: Lang *et al.* (2000); Wang *et al.* (1999*a,b*).



Experimental

Crystal data

$\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot \text{Cl}_2\text{I}^- \cdot \text{Cl}^-$
 $M_r = 295.37$
 Monoclinic, $C2/c$
 $a = 8.565$ (2) Å
 $b = 16.2186$ (15) Å
 $c = 19.9631$ (16) Å
 $\beta = 101.164$ (16)°
 $V = 2720.8$ (7) Å³

$Z = 12$
 Mo $K\alpha$ radiation
 $\mu = 4.34$ mm⁻¹

$T = 293$ K
 $0.36 \times 0.30 \times 0.28$ mm

Data collection

Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.230$, $T_{\max} = 0.301$

13418 measured reflections
 3106 independent reflections
 2821 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.056$
 $S = 1.10$
 3106 reflections

114 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.92$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A...Cl1 ⁱ	0.89	2.65	3.410 (3)	144
N1—H1A...Cl3 ⁱ	0.89	2.76	3.341 (3)	124
N1—H1B...Cl4	0.89	2.27	3.136 (2)	164
N1—H1C...Cl5 ⁱ	0.89	2.27	3.148 (3)	168
N2—H2A...Cl4 ⁱⁱ	0.89	2.38	3.232 (3)	161
N2—H2B...Cl5 ⁱⁱⁱ	0.89	2.26	3.123 (3)	162
N2—H2C...Cl3 ⁱⁱ	0.89	2.40	3.246 (3)	159
N3—H3A...Cl3 ^{iv}	0.89	2.42	3.297 (2)	167
N3—H3B...Cl5	0.89	2.32	3.144 (3)	154
N3—H3C...Cl1 ⁱⁱ	0.89	2.49	3.319 (2)	155

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2588).

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supplementary materials

Acta Cryst. (2009). E65, o2625 [doi:10.1107/S1600536809039038]

Ethylenediamonium dichloriodide chloride

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Comment

Recently much attention has been devoted to combining protonated aromatic nitrogen bases with halide or polyhalide ions due to their interesting structural features (Tucker & Kroon, 1973; Bandoli *et al.*, 1978). In our laboratory, a compound containing diprotonated ethylenediamine and ICl_2 anions has been synthesized, its crystal structure is reported herein.

The asymmetric unit of the title compound, $[\text{C}_2\text{H}_{10}\text{N}_2]^{2+} \cdot [\text{ICl}_2]^- \cdot \text{Cl}^-$, (Fig. 1) consists of two diprotonated ethylenediamonium cations, two $[\text{ICl}_2]^-$ anions and two Cl^- anions. The dichloriodide anion $\text{Cl11}-\text{I1}-\text{Cl11A}$ has site symmetry 2 and is linear with $\text{Cl11}-\text{I1}-\text{Cl11A}$ bond angle of 179.55 (4). The $\text{Cl11}-\text{I1}$ bond length is similar to the values of 2.5417 (11) to 2.5575 (10) Å reported by (Wang *et al.*, 1999*a,b*). In $\text{Cl12}-\text{I2}-\text{Cl13}$ anion, the $\text{I2}-\text{Cl13}$ bond length of 2.6790 (9) Å is longer than $\text{I2}-\text{Cl12}$ bond length of 2.4518 (10) Å. The $\text{Cl12}-\text{I2}-\text{Cl13}$ is also nearly linear, the $\text{Cl12}-\text{I2}-\text{Cl13}$ bond angle being 178.30 (3)°. The nearly linear $\text{Cl}-\text{I}-\text{Cl}$ bonds are similar to those reported by Lang *et al.* (2000) and Wang *et al.* (1999*a,b*). The two independent cations show the different conformations, the N-C-C-N torsion angles being 160.1 (2) and -73.1 (4)°. The crystal structure is stabilized by intermolecular N—H...Cl hydrogen bonds (Fig. 2).

Experimental

KI (0.33 g) and I_2 (0.5 g) were dissolved in a mixed solution of ethanol (30 ml) and concentrated hydrochloric acid (10 ml, 36%). On addition of ethylenediamine (0.60 g) to the above solution, the mixture was stirred for 2 h, then filtered. The filtrate was left at room temperature to allow the solvent to evaporate. Yellow transparent block crystals were obtained after two weeks.

Refinement

H atoms were placed in calculated positions with C—H = 0.97 Å and N—H = 0.89 Å, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{N})$.

Figures

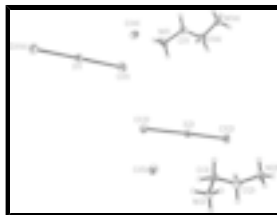


Fig. 1. The structure of the title compound with atom labels. Displacement ellipsoids were drawn at the 40% probability level [symmetry code: (i) $-x, y, -z+1/2$; (ii) $2-x, y, -z+1/2$].

Ethylenediammonium dichloriodide chloride

Crystal data

$C_2H_{10}N_2^{2+} \cdot Cl_2I^- \cdot Cl^-$	$F_{000} = 1680$
$M_r = 295.37$	$D_x = 2.163 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: $-C 2yc$	Cell parameters from 2821 reflections
$a = 8.565 (2) \text{ \AA}$	$\theta = 2.5\text{--}27.5^\circ$
$b = 16.2186 (15) \text{ \AA}$	$\mu = 4.34 \text{ mm}^{-1}$
$c = 19.9631 (16) \text{ \AA}$	$T = 293 \text{ K}$
$\beta = 101.164 (16)^\circ$	Block, yellow
$V = 2720.8 (7) \text{ \AA}^3$	$0.36 \times 0.30 \times 0.28 \text{ mm}$
$Z = 12$	

Data collection

Rigaku SCXmini diffractometer	3106 independent reflections
Radiation source: fine-focus sealed tube	2821 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.034$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 293 \text{ K}$	$\theta_{\text{min}} = 2.5^\circ$
ω scans	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$k = -20 \rightarrow 20$
$T_{\text{min}} = 0.230$, $T_{\text{max}} = 0.301$	$l = -25 \rightarrow 25$
13418 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.023$	$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2]$
$wR(F^2) = 0.056$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3106 reflections	$\Delta\rho_{\text{max}} = 0.92 \text{ e \AA}^{-3}$
114 parameters	$\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: $0.00017 (4)$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9115 (3)	0.1716 (2)	0.24029 (15)	0.0472 (8)
H1D	0.8758	0.2243	0.2199	0.057*
H1E	0.8778	0.1289	0.2065	0.057*
C2	1.0658 (4)	0.8169 (2)	0.43343 (16)	0.0412 (7)
H2D	1.0988	0.8492	0.4747	0.049*
H2E	1.1129	0.7626	0.4416	0.049*
C3	0.8881 (4)	0.80844 (18)	0.41979 (16)	0.0415 (7)
H3D	0.8539	0.7849	0.3747	0.050*
H3E	0.8592	0.7700	0.4526	0.050*
C11	0.30378 (10)	0.45106 (5)	0.26597 (4)	0.04654 (19)
C12	1.16812 (9)	0.58195 (6)	0.42064 (4)	0.04530 (19)
C13	0.57256 (8)	0.54598 (5)	0.41952 (3)	0.03576 (16)
C14	0.5000	0.23483 (7)	0.2500	0.0401 (2)
C15	0.47720 (9)	0.79063 (5)	0.40942 (4)	0.04283 (18)
I1	0.0000	0.450435 (17)	0.2500	0.03375 (8)
I2	0.88263 (2)	0.564990 (11)	0.418170 (9)	0.02956 (7)
N1	0.8379 (3)	0.15711 (16)	0.29954 (12)	0.0395 (6)
H1A	0.8485	0.1042	0.3114	0.059*
H1B	0.7351	0.1700	0.2890	0.059*
H1C	0.8855	0.1883	0.3342	0.059*
N2	1.1282 (3)	0.85669 (15)	0.37681 (12)	0.0384 (6)
H2A	1.0820	0.8342	0.3372	0.058*
H2B	1.2330	0.8492	0.3832	0.058*
H2C	1.1069	0.9104	0.3762	0.058*
N3	0.8007 (3)	0.88669 (15)	0.42352 (12)	0.0382 (6)
H3A	0.8401	0.9123	0.4626	0.057*
H3B	0.6981	0.8759	0.4216	0.057*
H3C	0.8115	0.9189	0.3886	0.057*

Atomic displacement parameters (\AA^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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supplementary materials

C1	0.0272 (16)	0.084 (3)	0.0303 (15)	-0.0052 (16)	0.0042 (13)	0.0027 (16)
C2	0.0345 (17)	0.0478 (18)	0.0391 (16)	0.0067 (13)	0.0013 (13)	0.0026 (14)
C3	0.0399 (18)	0.0322 (16)	0.0529 (19)	-0.0026 (12)	0.0100 (15)	0.0020 (14)
C11	0.0356 (4)	0.0628 (5)	0.0405 (4)	0.0103 (4)	0.0056 (3)	0.0010 (4)
C12	0.0291 (4)	0.0586 (5)	0.0487 (4)	-0.0054 (3)	0.0089 (3)	0.0000 (4)
C13	0.0263 (3)	0.0409 (4)	0.0391 (4)	0.0016 (3)	0.0038 (3)	-0.0005 (3)
C14	0.0340 (5)	0.0407 (6)	0.0420 (6)	0.000	-0.0016 (4)	0.000
C15	0.0300 (4)	0.0488 (4)	0.0488 (4)	-0.0043 (3)	0.0055 (3)	-0.0040 (3)
I1	0.03698 (16)	0.03827 (15)	0.02565 (13)	0.000	0.00517 (11)	0.000
I2	0.02793 (11)	0.03197 (11)	0.02788 (10)	0.00081 (7)	0.00317 (7)	-0.00044 (7)
N1	0.0337 (13)	0.0450 (15)	0.0410 (14)	0.0042 (11)	0.0099 (11)	0.0038 (11)
N2	0.0295 (13)	0.0401 (14)	0.0463 (14)	0.0023 (10)	0.0090 (11)	-0.0025 (11)
N3	0.0310 (13)	0.0435 (14)	0.0413 (14)	-0.0039 (11)	0.0103 (11)	-0.0044 (11)

Geometric parameters (\AA , $^\circ$)

C1—N1	1.463 (4)	C12—I2	2.4518 (10)
C1—C1 ⁱ	1.491 (6)	C13—I2	2.6790 (9)
C1—H1D	0.9700	I1—C11 ⁱⁱ	2.5595 (10)
C1—H1E	0.9700	N1—H1A	0.8900
C2—N2	1.488 (4)	N1—H1B	0.8900
C2—C3	1.499 (4)	N1—H1C	0.8900
C2—H2D	0.9700	N2—H2A	0.8900
C2—H2E	0.9700	N2—H2B	0.8900
C3—N3	1.483 (4)	N2—H2C	0.8900
C3—H3D	0.9700	N3—H3A	0.8900
C3—H3E	0.9700	N3—H3B	0.8900
C11—I1	2.5595 (10)	N3—H3C	0.8900
N1—C1—C1 ⁱ	111.4 (3)	C12—I2—C13	178.30 (3)
N1—C1—H1D	109.3	C1—N1—H1A	109.5
C1 ⁱ —C1—H1D	109.3	C1—N1—H1B	109.5
N1—C1—H1E	109.3	H1A—N1—H1B	109.5
C1 ⁱ —C1—H1E	109.3	C1—N1—H1C	109.5
H1D—C1—H1E	108.0	H1A—N1—H1C	109.5
N2—C2—C3	113.8 (3)	H1B—N1—H1C	109.5
N2—C2—H2D	108.8	C2—N2—H2A	109.5
C3—C2—H2D	108.8	C2—N2—H2B	109.5
N2—C2—H2E	108.8	H2A—N2—H2B	109.5
C3—C2—H2E	108.8	C2—N2—H2C	109.5
H2D—C2—H2E	107.7	H2A—N2—H2C	109.5
N3—C3—C2	114.7 (3)	H2B—N2—H2C	109.5
N3—C3—H3D	108.6	C3—N3—H3A	109.5
C2—C3—H3D	108.6	C3—N3—H3B	109.5
N3—C3—H3E	108.6	H3A—N3—H3B	109.5
C2—C3—H3E	108.6	C3—N3—H3C	109.5
H3D—C3—H3E	107.6	H3A—N3—H3C	109.5
C11—I1—C11 ⁱⁱ	179.55 (4)	H3B—N3—H3C	109.5

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

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—H1A···C11 ⁱⁱⁱ	0.89	2.65	3.410 (3)	144
N1—H1A···C13 ⁱⁱⁱ	0.89	2.76	3.341 (3)	124
N1—H1B···C14	0.89	2.27	3.136 (2)	164
N1—H1C···C15 ⁱⁱⁱ	0.89	2.27	3.148 (3)	168
N2—H2A···C14 ^{iv}	0.89	2.38	3.232 (3)	161
N2—H2B···C15 ^v	0.89	2.26	3.123 (3)	162
N2—H2C···C13 ^{iv}	0.89	2.40	3.246 (3)	159
N3—H3A···C13 ^{vi}	0.89	2.42	3.297 (2)	167
N3—H3B···C15	0.89	2.32	3.144 (3)	154
N3—H3C···C11 ^{iv}	0.89	2.49	3.319 (2)	155

Symmetry codes: (iii) $x+1/2, y-1/2, z$; (iv) $x+1/2, y+1/2, z$; (v) $x+1, y, z$; (vi) $-x+3/2, -y+3/2, -z+1$.

Fig. 1

