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Ethylenediammonium chloride thiocyanate

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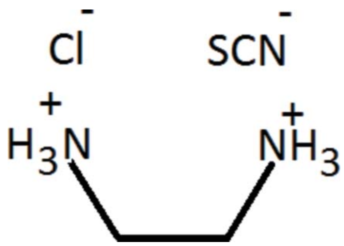
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.072; data-to-parameter ratio = 29.6.

In the ethylenediammonium dication of the title salt, $C_2H_{10}N_2^{2+}\cdot Cl^-\cdot SCN^-$, the N—C—C—N torsion angle is 72.09 (12)°. In the crystal, an extensive three-dimensional hydrogen-bonding network, formed by N—H⋯Cl and N—H⋯N hydrogen bonds, holds all the ions together.

Related literature

For the crystal structures of related compounds, see: Kamoun *et al.* (1989); Chen (2009). For details of the synthesis of thiocyanic acid, see: Bartlett *et al.* (1969). For protonic conductivity and dielectric relaxation in ethylendiammonium salts, see: Karoui *et al.* (2013).



Experimental

Crystal data

$C_2H_{10}N_2^{2+}\cdot Cl^-\cdot SCN^-$
M_r = 155.65
Triclinic, $P\overline{1}$
a = 6.2726 (2) Å
b = 6.3462 (2) Å
c = 9.1745 (3) Å
α = 92.436 (3)°
β = 92.193 (3)°
γ = 94.341 (3)°
V = 363.52 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.72 mm^{-1}
T = 293 K
0.50 × 0.42 × 0.17 mm

Data collection

Agilent Xcalibur (Sapphire2) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min} = 0.737, T_{max} = 0.887
6396 measured reflections
2189 independent reflections
1947 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.072
S = 1.08
2189 reflections
74 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯Cl1^{i}	0.89	2.36	3.1982 (9)	158
N2—H2B⋯Cl1^{ii}	0.89	2.35	3.2246 (10)	169
N2—H2C⋯Cl1^{iii}	0.89	2.64	3.3237 (10)	134
N3—H3C⋯Cl1^{iii}	0.89	2.46	3.2953 (11)	158
N3—H3A⋯N1^{iv}	0.89	2.03	2.8533 (14)	153
N2—H2C⋯Cl1	0.89	2.64	3.3543 (10)	138
N3—H3B⋯N1	0.89	2.27	3.1106 (17)	157

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg *et al.*, 1999) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5396).

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supporting information

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Sahel Karoui, Slaheddine Kamoun and François Michaud

S1. Comment

As an extension of our earlier study on protonic conductivity and dielectric relaxation in ethylenediammonium salts (Karoui *et al.*, 2013), we report herein the molecular structure of the title compound, (I), which is a new organic halide-pseudohalide compound.

In (I) (Fig. 1), the asymmetric unit consists of one diprotonated ethylenediammonium cation, one Cl^- and one SCN^- anions. In this atomic arrangement, the organic group has no internal symmetry. In fact, the mean length of the C—N bonds: 1.4816 (14) Å is lower than that of the C—C bonds:1.5054 (15) Å. The $[C_2H_{10}N_2]^{2+}$ dication shows an eclipsed conformation with a N–C–C–N torsion angle of 72.09 (12)°. The main geometrical features of this group are similar to that reported for others ethylenediammonium halides (Chen, 2009) and phosphates (Kamoun *et al.*,1989). The thiocyanate ion, present as a monodentate ligand, is almost linear with an angle of 178.48 (11)° and an average C–S and C–N bond lengths of 1.6358 (12) Å and 1.1573 (16) Å, respectively.

In the crystal structure, the ethylenediammonium cations are linked to the chloride and thiocyanate anions by means of five medium N—H···Cl and two weak N—H···N(CS) intermolecular hydrogen bonds (Table 1) to form a three-dimensional network (Fig. 2). The N···Cl and N···N(CS) distances range from 3.1982 (9) to 3.3543 (10) Å and 2.8533 (14) Å to 3.1106 (17) Å, respectively. The sum of Van der Waal's radii of N and Cl, and N and O are 3.3 Å and 2.9 Å, respectively.

S2. Experimental

The title compound has been obtained as crystalline solid in the reaction of ethylenediamine with an aqueous acidic mixture of hydrochloric acid and thiocyanic acid (1/1 ratio). Thiocyanic acid was prepared using the published procedure (Bartlett *et al.*, 1969). After a slow solvent evaporation yellow crystals suitable for X-ray analysis were obtained.They were washed with diethyl ether and dried over P_2O_5.

S3. Refinement

The H atoms were positioned geometrically (the C—H and N—H bonds were respectively fixed at 0.96 and 0.89), and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.2 U_{eq}(C, N)$.

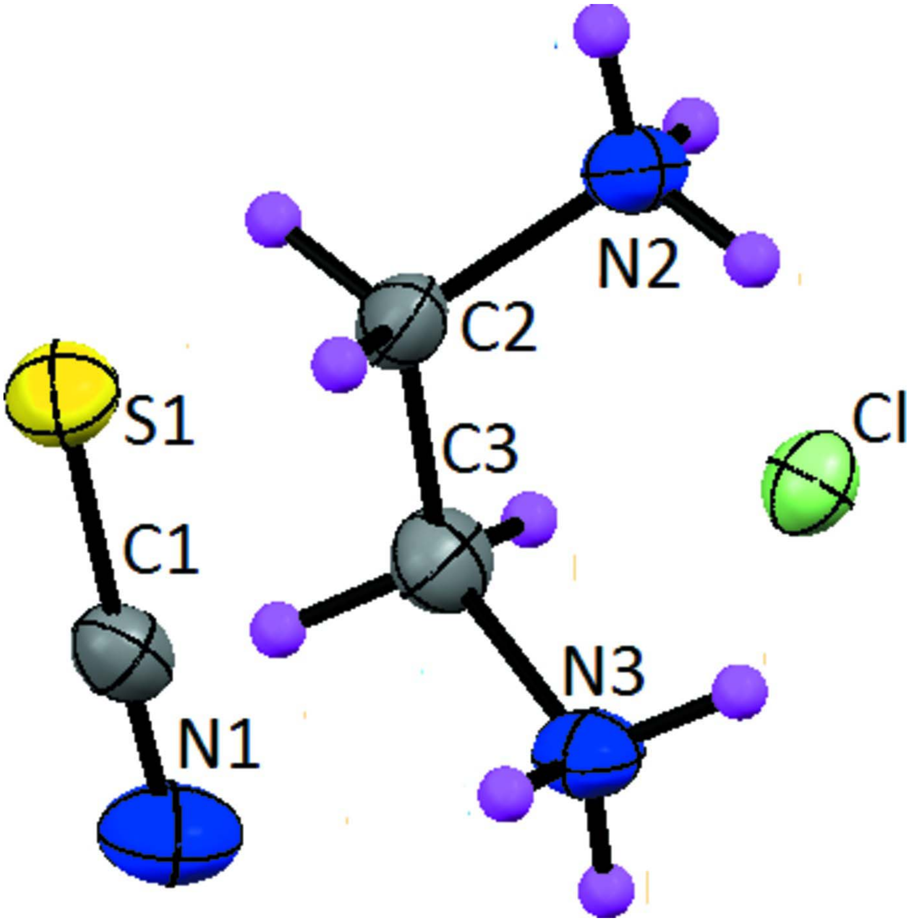

Figure 1

A content of asymmetric unit of (I) showing the atomic-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms represented by small spheres of arbitrary radii.

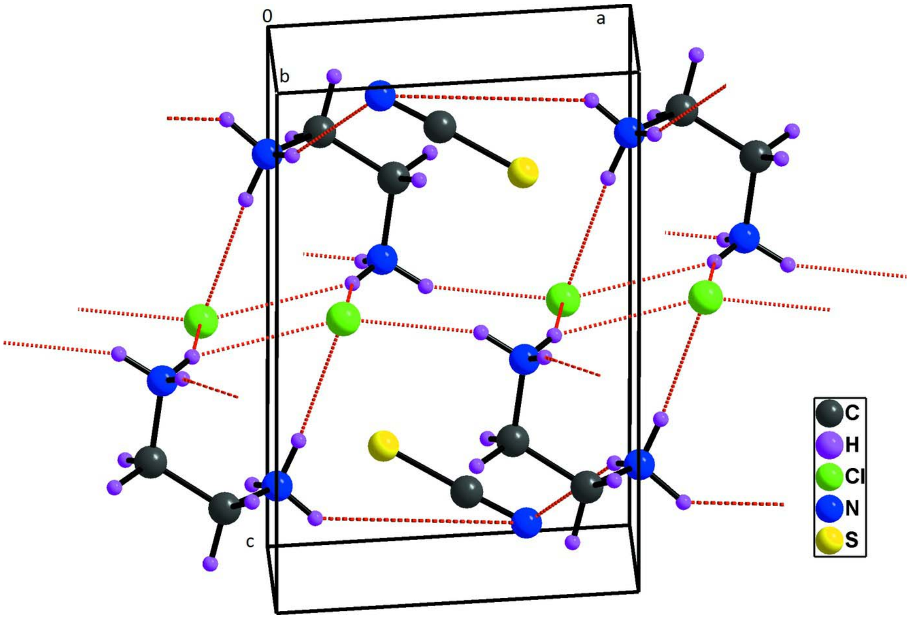

Figure 2

A portion of the crystal packing viewed approximately down the *b* axis and showing hydrogen bonds as dashed lines.

Ethylendiammonium chloride thiocyanate

Crystal data

$C_2H_{10}N_2^{2+}\cdot Cl^-\cdot SCN^-$
M_r = 155.65
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.2726 (2) Å
b = 6.3462 (2) Å
c = 9.1745 (3) Å
α = 92.436 (3)°
β = 92.193 (3)°
γ = 94.341 (3)°
V = 363.52 (2) Å^3
Z = 2
F(000) = 164

Cell parameters from 3445 reflections
D_x = 1.422 Mg m^{-3}
D_m = 1.398 Mg m^{-3}
D_m measured by flotation
Melting point: 443 K
Mo $K\alpha$ radiation, λ = 0.7107 Å
Cell parameters from 5534 reflections
θ = 3.2–44.7°
μ = 0.72 mm^{-1}
T = 293 K
Parallelipipedic, light yellow
0.50 × 0.42 × 0.17 mm

Data collection

Agilent Xcalibur (Sapphire2) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.3622 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min} = 0.737, T_{max} = 0.887

6396 measured reflections
2189 independent reflections
1947 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 30.5°, θ_{min} = 3.2°
$h = -5\rightarrow 8$
$k = -9\rightarrow 9$
$l = -13\rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H-atom parameters constrained
$wR(F^2) = 0.072$	$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.0716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
2189 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
74 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 constraints	Extinction coefficient: 0.294 (15)
Primary atom site location: structure-invariant direct methods	

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_{iso}*/U_{eq}
Cl1	0.19093 (4)	0.72470 (4)	0.47679 (3)	0.02934 (10)
S1	0.68519 (5)	0.78130 (5)	0.20850 (3)	0.03649 (10)
C1	0.45634 (19)	0.73609 (17)	0.11677 (11)	0.0294 (2)
N1	0.2924 (2)	0.7024 (2)	0.05464 (13)	0.0458 (3)
N2	0.31015 (15)	0.22026 (15)	0.43178 (10)	0.0305 (2)
H2A	0.4348	0.2481	0.4811	0.046*
H2B	0.2590	0.0888	0.4473	0.046*
H2C	0.2178	0.3109	0.4619	0.046*
C2	0.34163 (16)	0.24184 (17)	0.27393 (12)	0.0288 (2)
H2D	0.4449	0.1449	0.2424	0.035*
H2E	0.4007	0.3844	0.2582	0.035*
C3	0.13799 (19)	0.19753 (19)	0.18191 (12)	0.0322 (2)
H3E	0.1733	0.1811	0.0803	0.039*
H3D	0.0665	0.0651	0.2095	0.039*
N3	−0.01089 (16)	0.36715 (17)	0.19736 (11)	0.0345 (2)
H3A	−0.1283	0.3334	0.1412	0.052*
H3B	0.0525	0.4885	0.1701	0.052*
H3C	−0.0462	0.3812	0.2901	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02482 (13)	0.02877 (14)	0.03390 (15)	−0.00093 (8)	0.00252 (9)	−0.00075 (9)

S1	0.02975 (16)	0.04342 (18)	0.03524 (17)	−0.00037 (11)	−0.00273 (11)	−0.00101 (12)
C1	0.0356 (5)	0.0265 (5)	0.0262 (5)	0.0033 (4)	−0.0003 (4)	0.0020 (3)
N1	0.0446 (6)	0.0482 (6)	0.0434 (6)	0.0040 (5)	−0.0144 (5)	0.0008 (5)
N2	0.0269 (4)	0.0339 (5)	0.0312 (4)	0.0058 (3)	−0.0035 (3)	0.0048 (3)
C2	0.0229 (4)	0.0312 (5)	0.0329 (5)	0.0040 (4)	0.0042 (4)	0.0030 (4)
C3	0.0337 (5)	0.0347 (5)	0.0275 (5)	0.0027 (4)	−0.0009 (4)	−0.0066 (4)
N3	0.0291 (4)	0.0447 (5)	0.0296 (5)	0.0075 (4)	−0.0064 (3)	0.0002 (4)

Geometric parameters (Å, º)

S1—C1	1.6358 (12)	C2—H2E	0.9700
C1—N1	1.1573 (16)	C3—N3	1.4834 (15)
N2—C2	1.4798 (14)	C3—H3E	0.9700
N2—H2A	0.8900	C3—H3D	0.9700
N2—H2B	0.8900	N3—H3A	0.8900
N2—H2C	0.8900	N3—H3B	0.8900
C2—C3	1.5054 (15)	N3—H3C	0.8900
C2—H2D	0.9700		
N1—C1—S1	178.48 (11)	N3—C3—C2	112.98 (9)
C2—N2—H2A	109.5	N3—C3—H3E	109.0
C2—N2—H2B	109.5	C2—C3—H3E	109.0
H2A—N2—H2B	109.5	N3—C3—H3D	109.0
C2—N2—H2C	109.5	C2—C3—H3D	109.0
H2A—N2—H2C	109.5	H3E—C3—H3D	107.8
H2B—N2—H2C	109.5	C3—N3—H3A	109.5
N2—C2—C3	113.06 (9)	C3—N3—H3B	109.5
N2—C2—H2D	109.0	H3A—N3—H3B	109.5
C3—C2—H2D	109.0	C3—N3—H3C	109.5
N2—C2—H2E	109.0	H3A—N3—H3C	109.5
C3—C2—H2E	109.0	H3B—N3—H3C	109.5
H2D—C2—H2E	107.8		
N2—C2—C3—N3	72.09 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1$^{\rm i}$	0.89	2.36	3.1982 (9)	158
N2—H2*B*···Cl1$^{\rm ii}$	0.89	2.35	3.2246 (10)	169
N2—H2*C*···Cl1$^{\rm iii}$	0.89	2.64	3.3237 (10)	134
N3—H3*C*···Cl1$^{\rm iii}$	0.89	2.46	3.2953 (11)	158
N3—H3*A*···N1$^{\rm iv}$	0.89	2.03	2.8533 (14)	153
N2—H2*C*···Cl1	0.89	2.64	3.3543 (10)	138
N3—H3*B*···N1	0.89	2.27	3.1106 (17)	157

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