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2-[Bis(2-aminoethyl)amino]-ethanaminium chloride dichloromethane solvate

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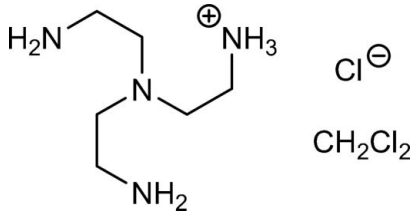
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.111; data-to-parameter ratio = 24.6.

In the title compound, $C_6H_{19}N_4^+\cdot Cl^-\cdot CH_2Cl_2$, the non-H atoms of the ammonium ion show non-crystallographic C_3 symmetry. The chloride ion is embedded in a framework of seven crystallographically independent hydrogen bonds (five N—H···Cl and two C—H···Cl), which form layers parallel to the (100) plane. Two N—H...N bonds also occur.

Related literature

For the crystal structure of *N*,*N*,*N*-tris(2-ammonioethyl)amine trichloride, see: Rasmussen & Hazell (1963); Hazell & Rasmussen (1968); Ilioudis *et al.* (2000).



Experimental

Crystal data

$C_6H_{19}N_4^+\cdot Cl^-\cdot CH_2Cl_2$
M_r = 267.63
Monoclinic, $P2_1/c$
a = 12.1512 (4) Å
b = 8.5686 (2) Å
c = 13.5497 (3) Å
β = 104.273 (2)°
V = 1367.23 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 200 (2) K
0.17 × 0.17 × 0.17 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
10639 measured reflections
3130 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.03
3130 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 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
N2—H1···Cl1	0.94	2.54	3.3897 (17)	151
N2—H2···Cl1^{i}	0.97	2.58	3.4402 (16)	148
N3—H3···Cl1^{ii}	0.88	2.57	3.4013 (16)	159
N3—H4···Cl1	0.96	2.55	3.4203 (17)	150
N4—H5···Cl1	0.90	2.42	3.2562 (15)	153
N4—H6···N3^{iii}	0.93	1.96	2.862 (2)	165
N4—H7···N2^{iv}	1.02	1.73	2.746 (2)	171
C4—H14···Cl1^{iii}	0.99	2.82	3.7014 (18)	149
C7—H21···Cl1^{i}	0.99	2.54	3.482 (3)	160

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

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); 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: *SHELXL97*, *PLATON* (Spek, 200) and *Mercury* (Macrae *et al.*, 2006).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2162).

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supporting information

Acta Cryst. (2008). E**64**, o2500 [doi:10.1107/S1600536808039652]

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S1. Comment

The title compounds molecular structure is shown in Fig. 1. The ammonium ion does not exhibit any crystallographic symmetry but, excluding the hydrogen atoms, it shows non-crystallographic C_3 symmetry.

It has to be assumed that the chloride ion is formed by a nucleophilic substitution reaction between a part of the tris(2-aminoethyl)amine and the solvent dichloromethane.

In the crystal structure, hydrogen bonds between the ammonium and chloride ions and the solvate molecule form two-dimensional networks parallel to the (100) plane (see Fig. 2). The chloride ion is embedded in a framework of seven crystallographically independent hydrogen bonds (see Fig. 3).

S2. Experimental

Crystals of the title compound were obtained from a solution of tris(2-aminoethyl)amine (0.15 g, 5.0 mmol) and trimethylborate (0.52 g, 5.0 mmol) in dichloromethane (10 ml) upon slow evaporation of the solvent at room temperature.

S3. Refinement

All H atoms were found in difference maps. C-bonded H atoms were positioned geometrically (C—H = 0.99 Å) and treated as riding on their parent atoms [U_{iso}(H) = 1.2U_{eq}(C)]. N-bonded H atoms were assigned from difference maps and treated as riding on their parent atoms [U_{iso}(H) = 1.2U_{eq}(N)].

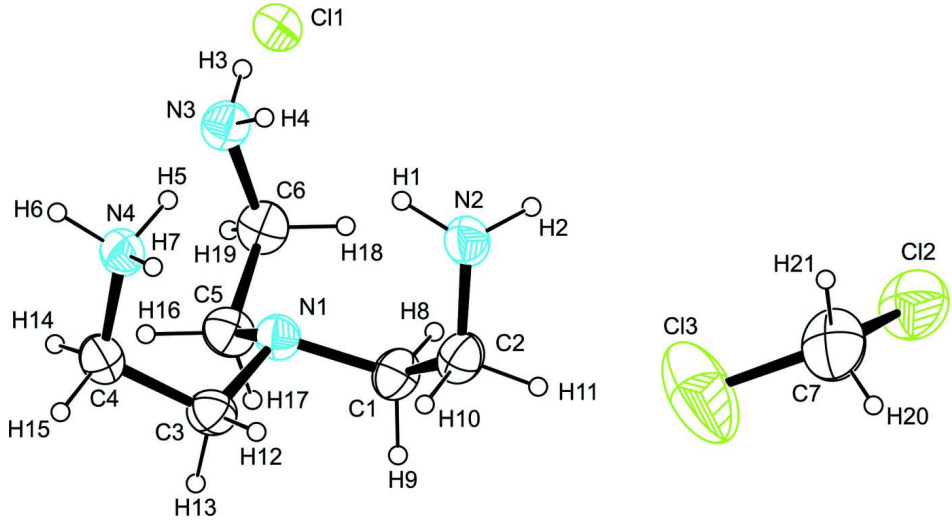

Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

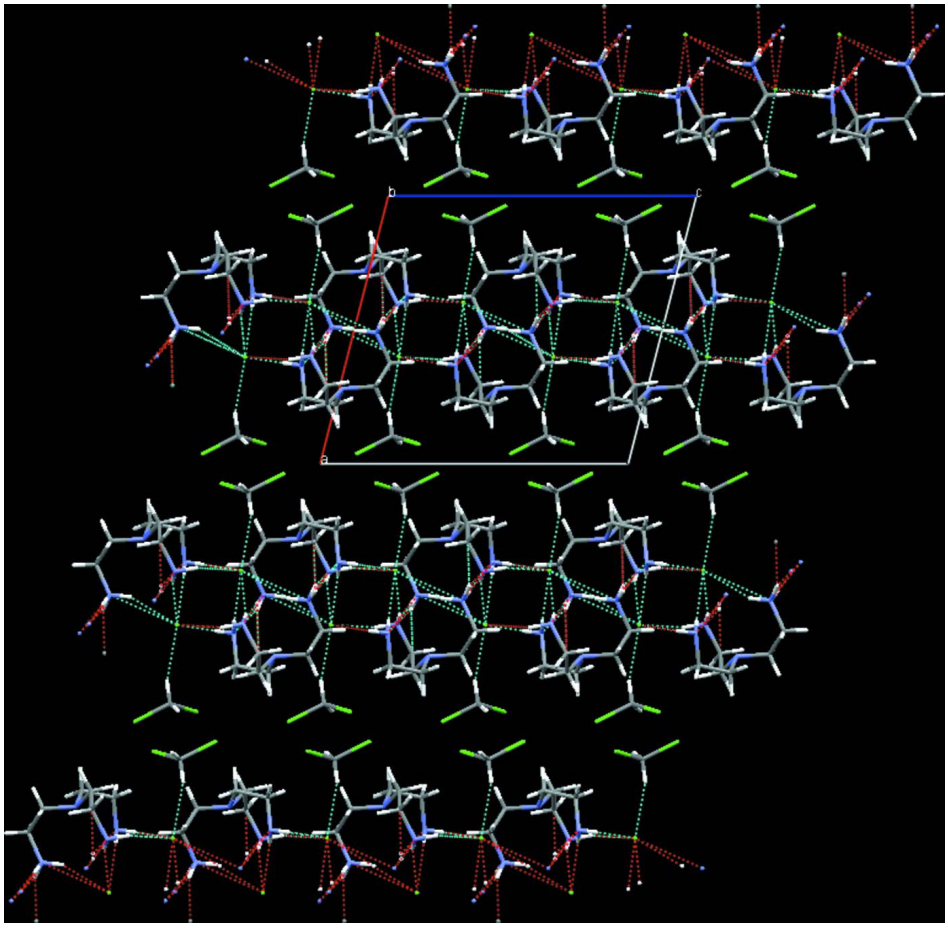

Figure 2

The packing and the hydrogen-bonded layers in the title compound, viewed along [0 1 0].

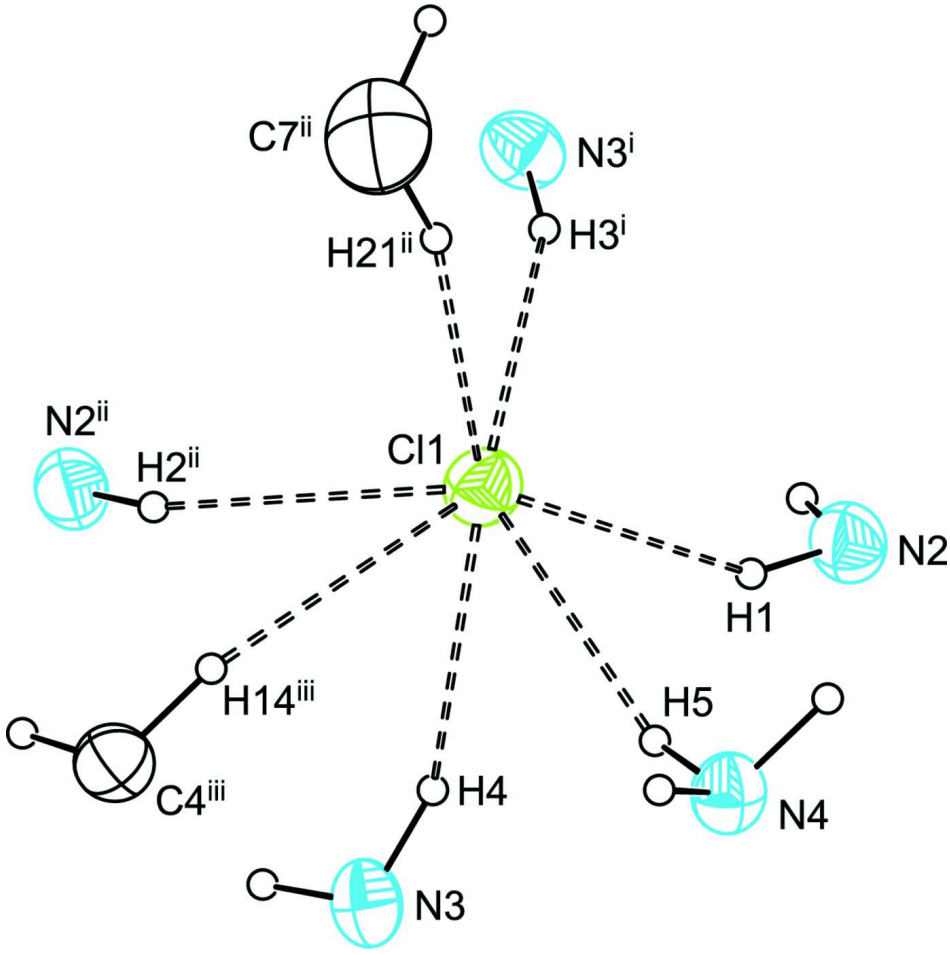

Figure 3

Hydrogen bonding to Cl1. [Symmetry codes: (i) 1 - *x*, 1/2 + *y*, 1/2 - *z*; (ii) 1 - *x*, -1/2 + *y*, 1/2 - *z*; (iii) 1 - *x*, -*y*, -*z*.]

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Crystal data

$C_6H_{19}N_4^+\cdot Cl^-\cdot CH_2Cl_2$
$M_r = 267.63$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.1512\ (4)$ Å
$b = 8.5686\ (2)$ Å
$c = 13.5497\ (3)$ Å
$\beta = 104.273\ (2)°$
$V = 1367.23\ (6)$ Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.300$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 17190 reflections
$\theta = 3.1–27.5°$
$\mu = 0.65$ mm^{-1}
$T = 200$ K
Block, colourless
0.17 × 0.17 × 0.17 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: rotating anode
MONTEL, graded multilayered X-ray optics monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans
10639 measured reflections
3130 independent reflections
2431 reflections with $I > 2\sigma(I)$

R_{int} = 0.030
θ_{max} = 27.5°, θ_{min} = 3.2°
h = −15→15
k = −11→10
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.03
3130 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.7878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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 $F^2 > 2\sigma(F^2)$ 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}
N1	0.28042 (13)	0.26045 (16)	−0.00985 (11)	0.0291 (3)
N2	0.40189 (14)	0.52948 (18)	0.11822 (12)	0.0355 (4)
H1	0.4350	0.4312	0.1169	0.043*
H2	0.3888	0.5495	0.1846	0.043*
N3	0.36750 (14)	0.01291 (18)	0.14123 (12)	0.0347 (4)
H3	0.3757	−0.0326	0.2006	0.042*
H4	0.4111	0.1080	0.1487	0.042*
N4	0.48946 (13)	0.20788 (18)	−0.07457 (11)	0.0324 (3)
H5	0.4973	0.2055	−0.0066	0.039*
H6	0.5435	0.1373	−0.0843	0.039*
H7	0.5240	0.3119	−0.0890	0.039*
C1	0.22269 (16)	0.3910 (2)	0.02691 (15)	0.0362 (4)
H8	0.2052	0.3602	0.0918	0.043*
H9	0.1497	0.4113	−0.0232	0.043*
C2	0.29173 (17)	0.5407 (2)	0.04382 (15)	0.0364 (4)
H10	0.3049	0.5746	−0.0222	0.044*
H11	0.2462	0.6228	0.0666	0.044*
C3	0.28825 (16)	0.2874 (2)	−0.11498 (13)	0.0342 (4)
H12	0.3085	0.3979	−0.1226	0.041*
H13	0.2132	0.2678	−0.1621	0.041*
C4	0.37594 (16)	0.1831 (2)	−0.14384 (13)	0.0343 (4)
H14	0.3534	0.0725	−0.1405	0.041*

H15	0.3792	0.2058	−0.2147	0.041*
C5	0.22065 (16)	0.1131 (2)	−0.00284 (14)	0.0355 (4)
H16	0.2424	0.0354	−0.0487	0.043*
H17	0.1378	0.1309	−0.0268	0.043*
C6	0.24633 (17)	0.0466 (2)	0.10427 (14)	0.0362 (4)
H18	0.2230	0.1224	0.1504	0.043*
H19	0.2022	−0.0505	0.1044	0.043*
Cl2	0.03789 (6)	0.73386 (9)	0.38860 (6)	0.0703 (2)
Cl3	0.07369 (8)	0.62211 (16)	0.19725 (8)	0.1163 (4)
C7	0.1040 (2)	0.7685 (3)	0.2895 (2)	0.0630 (7)
H20	0.0780	0.8702	0.2574	0.076*
H21	0.1871	0.7746	0.3178	0.076*
Cl1	0.60198 (4)	0.24526 (5)	0.16855 (3)	0.03439 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0323 (8)	0.0290 (7)	0.0272 (7)	−0.0003 (6)	0.0093 (6)	0.0019 (6)
N2	0.0442 (9)	0.0298 (8)	0.0320 (8)	0.0009 (7)	0.0082 (7)	−0.0019 (6)
N3	0.0409 (9)	0.0321 (8)	0.0333 (8)	0.0032 (7)	0.0132 (7)	0.0056 (6)
N4	0.0375 (8)	0.0331 (8)	0.0276 (7)	0.0026 (7)	0.0101 (6)	0.0001 (6)
C1	0.0329 (10)	0.0361 (10)	0.0412 (10)	0.0026 (8)	0.0123 (8)	−0.0008 (8)
C2	0.0398 (10)	0.0306 (9)	0.0403 (10)	0.0042 (8)	0.0127 (8)	0.0019 (8)
C3	0.0372 (10)	0.0371 (10)	0.0285 (9)	0.0024 (8)	0.0082 (8)	0.0051 (7)
C4	0.0394 (10)	0.0344 (10)	0.0296 (9)	−0.0016 (8)	0.0095 (8)	−0.0031 (7)
C5	0.0350 (10)	0.0347 (10)	0.0355 (9)	−0.0069 (8)	0.0065 (8)	0.0000 (8)
C6	0.0383 (10)	0.0352 (10)	0.0382 (10)	−0.0026 (8)	0.0151 (8)	0.0037 (8)
Cl2	0.0586 (4)	0.0835 (5)	0.0745 (4)	0.0079 (3)	0.0269 (3)	0.0044 (3)
Cl3	0.0780 (6)	0.1752 (11)	0.1012 (7)	−0.0436 (6)	0.0326 (5)	−0.0730 (7)
C7	0.0437 (13)	0.0711 (17)	0.0787 (18)	−0.0073 (12)	0.0239 (13)	−0.0116 (14)
Cl1	0.0357 (3)	0.0366 (3)	0.0302 (2)	−0.00045 (18)	0.00691 (17)	−0.00011 (17)

Geometric parameters (Å, º)

N1—C3	1.469 (2)	C2—H10	0.9900
N1—C1	1.471 (2)	C2—H11	0.9900
N1—C5	1.471 (2)	C3—C4	1.514 (3)
N2—C2	1.467 (2)	C3—H12	0.9900
N2—H1	0.9358	C3—H13	0.9900
N2—H2	0.9660	C4—H14	0.9900
N3—C6	1.462 (2)	C4—H15	0.9900
N3—H3	0.8776	C5—C6	1.518 (3)
N3—H4	0.9638	C5—H16	0.9900
N4—C4	1.480 (2)	C5—H17	0.9900
N4—H5	0.9019	C6—H18	0.9900
N4—H6	0.9265	C6—H19	0.9900
N4—H7	1.0244	Cl2—C7	1.751 (3)
C1—C2	1.519 (3)	Cl3—C7	1.745 (3)

C1—H8	0.9900	C7—H20	0.9900
C1—H9	0.9900	C7—H21	0.9900
C3—N1—C1	111.05 (14)	C4—C3—H12	109.2
C3—N1—C5	110.38 (14)	N1—C3—H13	109.2
C1—N1—C5	110.28 (14)	C4—C3—H13	109.2
C2—N2—H1	111.7	H12—C3—H13	107.9
C2—N2—H2	107.2	N4—C4—C3	110.89 (15)
H1—N2—H2	110.4	N4—C4—H14	109.5
C6—N3—H3	106.2	C3—C4—H14	109.5
C6—N3—H4	110.5	N4—C4—H15	109.5
H3—N3—H4	110.1	C3—C4—H15	109.5
C4—N4—H5	119.5	H14—C4—H15	108.0
C4—N4—H6	113.5	N1—C5—C6	113.30 (15)
H5—N4—H6	103.3	N1—C5—H16	108.9
C4—N4—H7	111.5	C6—C5—H16	108.9
H5—N4—H7	105.6	N1—C5—H17	108.9
H6—N4—H7	101.7	C6—C5—H17	108.9
N1—C1—C2	113.68 (15)	H16—C5—H17	107.7
N1—C1—H8	108.8	N3—C6—C5	110.71 (15)
C2—C1—H8	108.8	N3—C6—H18	109.5
N1—C1—H9	108.8	C5—C6—H18	109.5
C2—C1—H9	108.8	N3—C6—H19	109.5
H8—C1—H9	107.7	C5—C6—H19	109.5
N2—C2—C1	115.12 (15)	H18—C6—H19	108.1
N2—C2—H10	108.5	Cl3—C7—Cl2	111.83 (15)
C1—C2—H10	108.5	Cl3—C7—H20	109.2
N2—C2—H11	108.5	Cl2—C7—H20	109.2
C1—C2—H11	108.5	Cl3—C7—H21	109.2
H10—C2—H11	107.5	Cl2—C7—H21	109.2
N1—C3—C4	112.06 (15)	H20—C7—H21	107.9
N1—C3—H12	109.2		
C3—N1—C1—C2	−69.7 (2)	N1—C3—C4—N4	−58.4 (2)
C5—N1—C1—C2	167.57 (15)	C3—N1—C5—C6	158.47 (16)
N1—C1—C2—N2	−59.7 (2)	C1—N1—C5—C6	−78.5 (2)
C1—N1—C3—C4	162.77 (15)	N1—C5—C6—N3	−60.7 (2)
C5—N1—C3—C4	−74.60 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···Cl1	0.94	2.54	3.3897 (17)	151
N2—H2···Cl1^{i}	0.97	2.58	3.4402 (16)	148
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