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ISSN 2414-3146

Bis(4-amino-3,5-dichloropyridinium) tetrachloridomercurate(II)

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Received 3 December 2025
Accepted 11 December 2025

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

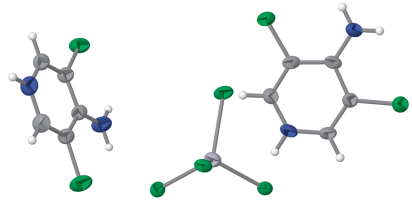
Keywords: crystal structure; hydrogen bonding; mercury.

CCDC reference: 2502353

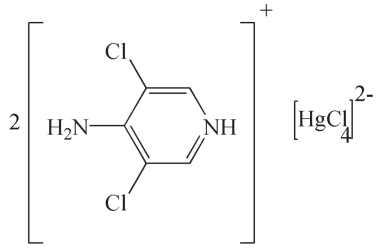
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $(C_5H_5Cl_2N_2)_2[HgCl_4]$, comprises two 4-amino-3,5-dichloropyridinium cations protonated at the pyridine N atoms and one tetrahedral tetrachloridomercurate(II) anion. The linking forces in the extended structure are predominantly N—H···Cl, bifurcated N—H···(Cl,Cl) and C—H···Cl hydrogen bonds, which connect the cations and anions into a three-dimensional network.

3D view



Chemical scheme



Structure description

Following previous reports on the crystal structure of the parent 4-amino-3,5-dichloropyridine (Anantheeswary *et al.*, 2024) and our recent work on 4-amino-3,5-dichloropyridinium 3-hydroxypicolinate monohydrate (Ashokan *et al.*, 2023), we now describe the synthesis and structural characterization of the title salt, $(C_5H_5Cl_2N_2)_2[HgCl_4]$.

The mercury(II) atom in the complex anion is coordinated by four chlorido ligands (Fig. 1) in a distorted tetrahedral shape (Table 1), which is typical behaviour for a d^{10} soft Lewis acid. The Hg—Cl bond lengths range from 2.485 (15) to 2.491 (14) Å, while Cl—Hg—Cl angles fall in the range 103.00 (5) to 121.6 (5)°. The 4-amino-3,5-dichloropyridinium cations are protonated at atoms N1 and N3 of the pyridine moiety. This protonation is evidenced by the increase in the internal angle C1—N1—C5 = 121.5 (5)° and C10—N3—C6 = 121.8 (5)° compared with the corresponding angle in neutral 4-amino-5,6-dichloro pyridine [116.4 (5)°] (Anantheeswary *et al.*, 2024). Otherwise, the geometrical data for the title compound are in good agreement with literature data (Jellali *et al.*, 2024). In the extended structure, the components are linked by numerous N—H···Cl, N—H···(Cl,Cl) and C—H···Cl (Table 2) hydrogen bonds as shown in Fig. 2,

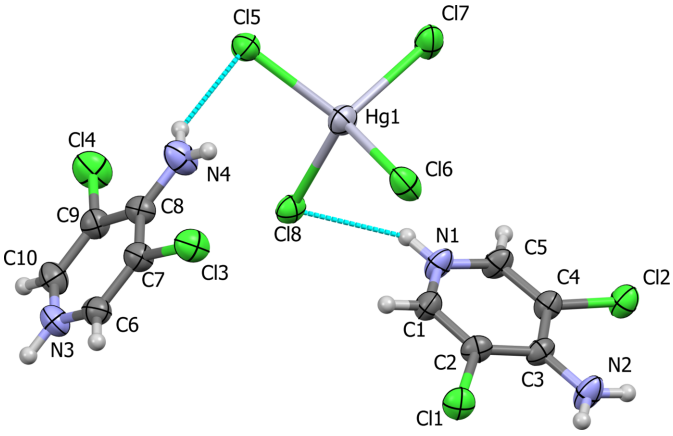


Figure 1
The asymmetric unit of the title compound with 50% probability displacement ellipsoids.

which generates a three-dimensional network. All the chlorido ions of the complex anion accept at least one hydrogen bond.

A search of the Cambridge Structural Database (Version 5.43, update November 2022; Groom *et al.*, 2016) for related structures revealed CSD refcodes BOLCUE: bis(3-amino-2-chloropyridinium)tetrakis(chlorido)mercurate(II) (Mrad *et al.*, 2024); AGEWUF: 2-2′-bipyridinediium tetrabromidomercurate(II) (Ali *et al.*, 2008); AJIKAG: bis(1,3-diethyl-1*H*-3,1-benzimidazole-3-ium)tetrabromidomercurate(II) (Li *et al.*, 2009) and AQEDUX: bis(3-{[(pyridine-3-yl)methyl]amino}-pyridine-1-ium) tetraiodidomercurate (Ye *et al.*, 2016).

Synthesis and crystallization

4-Amino-3,5-dichloropyridine (45 mg) and mercury(II) chloride (67 mg) were used as starting materials. Each reagent was dissolved separately in methanol and water, respectively. The solutions were combined and stirred magnetically at room temperature, with the addition of 2 to 3 drops of dilute HCl to get a clear solution.. The resulting clear solution was left to stand for slow evaporation at room temperature. Colourless crystals suitable for X-ray diffraction were obtained after several days.

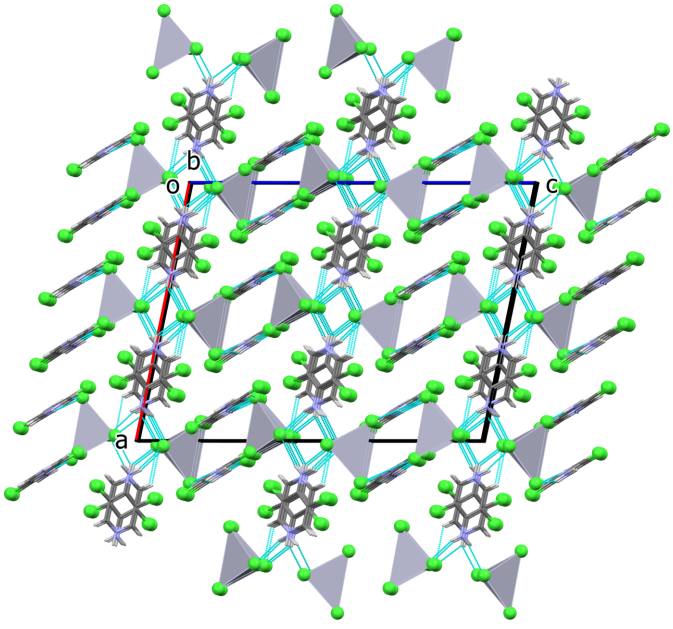


Figure 2
View of the crystal packing of the title compound along the *b*-axis direction with the complex anions shown in polyhedral representation.

Table 1
Selected geometric parameters (Å, °).

Hg1—Cl7	2.4771 (15)	Hg1—Cl6	2.4854 (15)
Hg1—Cl8	2.4846 (15)	Hg1—Cl5	2.4908 (14)
Cl7—Hg1—Cl8	121.61 (5)	Cl7—Hg1—Cl5	105.31 (5)
Cl7—Hg1—Cl6	103.00 (5)	Cl8—Hg1—Cl5	103.92 (5)
Cl8—Hg1—Cl6	104.09 (5)	Cl6—Hg1—Cl5	120.16 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Cl8	0.87	2.53	3.202 (6)	134
N1—H1*N*···Cl8^{i}	0.87	2.54	3.190 (6)	132
N2—H2*NA*···Cl7^{ii}	0.87	2.60	3.439 (5)	162
N2—H2*NB*···Cl7^{iii}	0.87	2.51	3.338 (5)	159
N3—H3*N*···Cl5^{iv}	0.87	2.78	3.387 (5)	128
N3—H3*N*···Cl6^{v}	0.87	2.60	3.255 (6)	133
N4—H4*NA*···Cl5	0.87	2.47	3.229 (5)	146
N4—H4*NB*···Cl6^{vi}	0.87	2.46	3.265 (6)	154
C1—H1···Cl8	0.94	2.81	3.356 (6)	118
C5—H5···Cl8^{i}	0.94	2.75	3.308 (6)	119
C10—H10···Cl5^{iv}	0.94	2.77	3.396 (6)	125

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (iii) $x+\frac{1}{2}, y+\frac{1}{2}, z$; (iv) $x, y+1, z$; (v) $-x+1, y+1, -z+\frac{1}{2}$; (vi) $-x+1, y, -z+\frac{1}{2}$.

Table 3
Experimental details.

Crystal data	
Chemical formula	$(C_5H_5Cl_2N_2)_2[HgCl_4]$
M_r	670.41
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	250
a, *b*, *c* (Å)	18.0721 (6), 9.2125 (2), 23.7047 (8)
β (°)	101.652 (3)
V (Å^3)	3865.2 (2)
Z	8
Radiation type	Mo *K*α
μ (mm^{-1})	9.07
Crystal size (mm)	0.76 × 0.54 × 0.34
Data collection	
Diffractometer	STOE *IPDS* II
Absorption correction	Multi-scan (*X-RED32*; Stoe, 2023)
T_{min}, T_{max}	0.035, 0.223
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19023, 3877, 3645
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.621
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.092, 1.13
No. of reflections	3877
No. of parameters	209
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.34, −1.43

Computer programs: *X-AREA WinXpose*, *Recipe*, *Integrate* and *LANA* (Stoe, 2023), *SHELXT2019/3* (Sheldrick, 2015*a*), *SHELXL2019/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 3.

Acknowledgements

HSE is grateful to the University of Neuchâtel for their support over the years.

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full crystallographic data

IUCrData (2025). **10**, x251120 [https://doi.org/10.1107/S2414314625011204]

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Crystal data

$(C_5H_5Cl_2N_2)_2[HgCl_4]$
M_r = 670.41
Monoclinic, *C*2/*c*
a = 18.0721 (6) Å
b = 9.2125 (2) Å
c = 23.7047 (8) Å
β = 101.652 (3)°
V = 3865.2 (2) Å^3
Z = 8
F(000) = 2512
D_x = 2.304 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 25660 reflections
θ = 1.8–26.8°
μ = 9.07 mm^{-1}
T = 250 K
Block, colourless
0.76 × 0.54 × 0.34 mm

Data collection

STOE IPDS II diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method, *ω* scans
Absorption correction: multi-scan (X-RED32; Stoe, 2023)
T_{min} = 0.035, T_{max} = 0.223
19023 measured reflections
3877 independent reflections
3645 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 26.2°, θ_{min} = 2.6°
h = −22→22
k = −11→10
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.092
S = 1.13
3877 reflections
209 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 32.4079P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.34 e Å^{-3}
$\Delta\rho_{min}$ = −1.43 e Å^{-3}
Extinction correction: (SHELXL-2019/3; Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00160 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were positioned geometrically [C—H = 0.94, N—H = 0.87 Å] and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(carrier).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.77457 (8)	0.70283 (17)	0.41102 (6)	0.0390 (3)
Cl2	0.82254 (8)	0.30420 (17)	0.58344 (6)	0.0400 (3)
N1	0.6440 (3)	0.5048 (6)	0.4991 (2)	0.0369 (11)
H1N	0.596006	0.505840	0.499608	0.044*
N2	0.8697 (3)	0.5031 (6)	0.4970 (2)	0.0430 (12)
H2NA	0.900317	0.448533	0.521073	0.052*
H2NB	0.887166	0.557362	0.472618	0.052*
C1	0.6698 (3)	0.5874 (6)	0.4607 (3)	0.0349 (12)
H1	0.635829	0.644304	0.434454	0.042*
C2	0.7442 (3)	0.5896 (6)	0.4594 (2)	0.0319 (12)
C3	0.7973 (3)	0.5036 (6)	0.4975 (2)	0.0304 (11)
C4	0.7652 (3)	0.4178 (6)	0.5361 (2)	0.0312 (11)
C5	0.6903 (3)	0.4209 (7)	0.5366 (2)	0.0352 (12)
H5	0.670954	0.364034	0.563185	0.042*
Cl3	0.45226 (9)	0.82260 (17)	0.15818 (7)	0.0444 (4)
Cl4	0.29236 (11)	0.8407 (2)	0.32800 (7)	0.0536 (4)
N3	0.3817 (3)	1.1382 (6)	0.2446 (2)	0.0403 (11)
H3N	0.384547	1.232482	0.245097	0.048*
N4	0.3683 (3)	0.6958 (6)	0.2426 (2)	0.0453 (13)
H4NA	0.344345	0.651024	0.265793	0.054*
H4NB	0.389316	0.646303	0.218772	0.054*
C6	0.4142 (3)	1.0637 (6)	0.2073 (3)	0.0365 (12)
H6	0.439799	1.113270	0.182342	0.044*
C7	0.4101 (3)	0.9163 (6)	0.2059 (3)	0.0343 (12)
C8	0.3727 (3)	0.8380 (6)	0.2434 (2)	0.0326 (11)
C9	0.3399 (3)	0.9243 (6)	0.2810 (3)	0.0349 (12)
C10	0.3449 (4)	1.0711 (6)	0.2810 (3)	0.0397 (13)
H10	0.322599	1.125959	0.306576	0.048*
Hg1	0.46587 (2)	0.40614 (3)	0.37445 (2)	0.03929 (12)
Cl5	0.33340 (8)	0.42430 (15)	0.31931 (6)	0.0376 (3)
Cl6	0.57310 (10)	0.42101 (15)	0.32352 (8)	0.0447 (4)
Cl7	0.47767 (8)	0.15743 (18)	0.41499 (7)	0.0447 (4)
Cl8	0.48132 (7)	0.62415 (16)	0.43745 (6)	0.0352 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0401 (7)	0.0461 (8)	0.0348 (7)	−0.0001 (6)	0.0173 (6)	0.0071 (6)
Cl2	0.0328 (7)	0.0520 (8)	0.0353 (7)	0.0013 (6)	0.0067 (5)	0.0099 (6)
N1	0.023 (2)	0.052 (3)	0.038 (3)	0.000 (2)	0.0127 (19)	0.003 (2)
N2	0.026 (2)	0.062 (3)	0.042 (3)	0.002 (2)	0.011 (2)	0.015 (2)
C1	0.027 (3)	0.042 (3)	0.035 (3)	0.001 (2)	0.005 (2)	0.004 (2)
C2	0.033 (3)	0.039 (3)	0.026 (3)	−0.005 (2)	0.013 (2)	−0.001 (2)
C3	0.025 (2)	0.041 (3)	0.028 (2)	−0.002 (2)	0.011 (2)	−0.005 (2)
C4	0.029 (3)	0.039 (3)	0.026 (3)	0.001 (2)	0.007 (2)	0.002 (2)
C5	0.029 (3)	0.049 (3)	0.030 (3)	−0.003 (2)	0.013 (2)	0.006 (2)
Cl3	0.0522 (9)	0.0443 (8)	0.0431 (8)	−0.0036 (7)	0.0250 (7)	−0.0067 (6)
Cl4	0.0700 (11)	0.0523 (9)	0.0481 (9)	−0.0009 (8)	0.0345 (8)	0.0042 (7)
N3	0.048 (3)	0.028 (2)	0.045 (3)	0.003 (2)	0.010 (2)	−0.002 (2)
N4	0.060 (3)	0.036 (3)	0.048 (3)	0.001 (2)	0.029 (3)	−0.002 (2)
C6	0.037 (3)	0.035 (3)	0.037 (3)	−0.002 (2)	0.006 (2)	0.001 (2)
C7	0.038 (3)	0.034 (3)	0.032 (3)	−0.001 (2)	0.010 (2)	−0.001 (2)
C8	0.037 (3)	0.034 (3)	0.027 (3)	−0.002 (2)	0.007 (2)	0.000 (2)
C9	0.037 (3)	0.037 (3)	0.032 (3)	0.001 (2)	0.011 (2)	0.001 (2)
C10	0.048 (3)	0.034 (3)	0.039 (3)	0.005 (3)	0.012 (3)	−0.004 (2)
Hg1	0.03396 (16)	0.04079 (17)	0.04560 (17)	0.00137 (9)	0.01388 (10)	0.00097 (9)
Cl5	0.0353 (7)	0.0351 (7)	0.0416 (7)	−0.0016 (5)	0.0060 (6)	0.0068 (6)
Cl6	0.0570 (9)	0.0323 (7)	0.0555 (9)	0.0049 (6)	0.0370 (8)	0.0050 (6)
Cl7	0.0339 (7)	0.0474 (8)	0.0567 (9)	0.0056 (6)	0.0182 (6)	0.0199 (7)
Cl8	0.0288 (6)	0.0424 (7)	0.0378 (7)	−0.0016 (5)	0.0152 (5)	−0.0010 (6)

Geometric parameters (Å, º)

Cl1—C2	1.719 (5)	N3—C10	1.342 (8)
Cl2—C4	1.720 (6)	N3—C6	1.344 (8)
N1—C5	1.337 (8)	N3—H3N	0.8700
N1—C1	1.340 (8)	N4—C8	1.312 (8)
N1—H1N	0.8700	N4—H4NA	0.8700
N2—C3	1.312 (7)	N4—H4NB	0.8700
N2—H2NA	0.8700	C6—C7	1.360 (8)
N2—H2NB	0.8700	C6—H6	0.9400
C1—C2	1.353 (8)	C7—C8	1.418 (8)
C1—H1	0.9400	C8—C9	1.411 (8)
C2—C3	1.418 (8)	C9—C10	1.355 (8)
C3—C4	1.418 (8)	C10—H10	0.9400
C4—C5	1.357 (8)	Hg1—Cl7	2.4771 (15)
C5—H5	0.9400	Hg1—Cl8	2.4846 (15)
Cl3—C7	1.719 (6)	Hg1—Cl6	2.4854 (15)
Cl4—C9	1.720 (6)	Hg1—Cl5	2.4908 (14)
C5—N1—C1	121.5 (5)	C8—N4—H4NA	120.0
C5—N1—H1N	119.2	C8—N4—H4NB	120.0

C1—N1—H1N	119.2	H4NA—N4—H4NB	120.0
C3—N2—H2NA	120.0	N3—C6—C7	119.8 (6)
C3—N2—H2NB	120.0	N3—C6—H6	120.1
H2NA—N2—H2NB	120.0	C7—C6—H6	120.1
N1—C1—C2	120.6 (5)	C6—C7—C8	121.6 (6)
N1—C1—H1	119.7	C6—C7—Cl3	119.2 (5)
C2—C1—H1	119.7	C8—C7—Cl3	119.2 (4)
C1—C2—C3	121.6 (5)	N4—C8—C9	122.7 (5)
C1—C2—Cl1	118.5 (5)	N4—C8—C7	122.2 (5)
C3—C2—Cl1	119.9 (4)	C9—C8—C7	115.0 (5)
N2—C3—C4	122.8 (5)	C10—C9—C8	121.8 (6)
N2—C3—C2	122.9 (5)	C10—C9—Cl4	119.2 (5)
C4—C3—C2	114.3 (5)	C8—C9—Cl4	119.0 (4)
C5—C4—C3	122.1 (5)	N3—C10—C9	120.1 (6)
C5—C4—Cl2	118.5 (4)	N3—C10—H10	120.0
C3—C4—Cl2	119.4 (4)	C9—C10—H10	120.0
N1—C5—C4	119.9 (5)	Cl7—Hg1—Cl8	121.61 (5)
N1—C5—H5	120.1	Cl7—Hg1—Cl6	103.00 (5)
C4—C5—H5	120.1	Cl8—Hg1—Cl6	104.09 (5)
C10—N3—C6	121.8 (5)	Cl7—Hg1—Cl5	105.31 (5)
C10—N3—H3N	119.1	Cl8—Hg1—Cl5	103.92 (5)
C6—N3—H3N	119.1	Cl6—Hg1—Cl5	120.16 (6)

C5—N1—C1—C2	−0.9 (9)	C10—N3—C6—C7	0.2 (9)
N1—C1—C2—C3	1.1 (9)	N3—C6—C7—C8	−0.8 (9)
N1—C1—C2—Cl1	−177.6 (5)	N3—C6—C7—Cl3	−179.8 (5)
C1—C2—C3—N2	179.7 (6)	C6—C7—C8—N4	−180.0 (6)
Cl1—C2—C3—N2	−1.6 (8)	Cl3—C7—C8—N4	−0.9 (8)
C1—C2—C3—C4	−0.1 (8)	C6—C7—C8—C9	1.0 (8)
Cl1—C2—C3—C4	178.5 (4)	Cl3—C7—C8—C9	180.0 (4)
N2—C3—C4—C5	179.2 (6)	N4—C8—C9—C10	−179.7 (6)
C2—C3—C4—C5	−1.0 (8)	C7—C8—C9—C10	−0.7 (9)
N2—C3—C4—Cl2	−1.7 (8)	N4—C8—C9—Cl4	0.4 (8)
C2—C3—C4—Cl2	178.2 (4)	C7—C8—C9—Cl4	179.4 (4)
C1—N1—C5—C4	−0.2 (9)	C6—N3—C10—C9	0.1 (10)
C3—C4—C5—N1	1.2 (9)	C8—C9—C10—N3	0.2 (10)
Cl2—C4—C5—N1	−178.0 (5)	Cl4—C9—C10—N3	−179.9 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Cl8	0.87	2.53	3.202 (6)	134
N1—H1*N*···Cl8^{i}	0.87	2.54	3.190 (6)	132
N2—H2*NA*···Cl7^{ii}	0.87	2.60	3.439 (5)	162
N2—H2*NB*···Cl7^{iii}	0.87	2.51	3.338 (5)	159
N3—H3*N*···Cl5^{iv}	0.87	2.78	3.387 (5)	128
N3—H3*N*···Cl6^{v}	0.87	2.60	3.255 (6)	133
N4—H4*NA*···Cl5	0.87	2.47	3.229 (5)	146

N4—H4*NB*···Cl6^{vi}	0.87	2.46	3.265 (6)	154
C1—H1···Cl8	0.94	2.81	3.356 (6)	118
C5—H5···Cl8^{i}	0.94	2.75	3.308 (6)	119
C10—H10···Cl5^{iv}	0.94	2.77	3.396 (6)	125

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+3/2, −*y*+1/2, −*z*+1; (iii) *x*+1/2, *y*+1/2, *z*; (iv) *x*, *y*+1, *z*; (v) −*x*+1, *y*+1, −*z*+1/2; (vi) −*x*+1, *y*, −*z*+1/2.