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N,*N*,*N*′,*N*′,*N*″,*N*″-Hexamethylguanidinium di-μ_3-chlorido-tetra-μ_2-chlorido-decachloridotetrabismuthate acetonitrile disolvate

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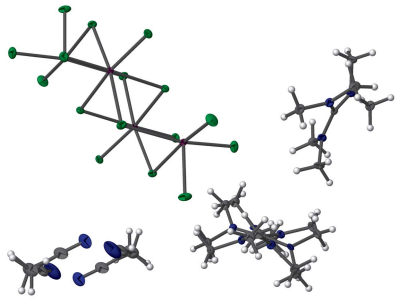
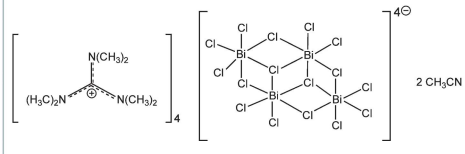
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the solvated title compound, $(C_7H_{18}N_3)_4[Bi_4Cl_{16}]\cdot 2CH_3CN$, comprises two cations, one half $[Bi_4Cl_{16}]^{4-}$ ion and one acetonitrile molecule. One *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium ion shows orientational disorder and two sets of N- and C-atom positions were found, with an occupancy ratio of 0.941 (2):0.059 (2). The second cation is not disordered. The C—N bond lengths in the two guanidinium ions range from 1.334 (17) to 1.341 (17) Å, indicating double-bond character and pointing towards charge delocalization within the NCN planes. The four Bi^{III} ions are coordinated by six chloride ions in distorted octahedral manner. Two $[Bi_2Cl_8]^{2-}$ dimers are fused together, forming a centrosymmetric tetranuclear $[Bi_4Cl_{16}]^{4-}$ cluster. The bond lengths of bismuth to the terminal chlorides [2.4982 (7)–2.5509 (6) Å] are shorter than those of the double and triply bridging ones [2.7052 (6)–3.0320 (6) Å]. The acetonitrile solvent molecule is disordered over two positions, with an occupancy ratio of 0.818 (4):0.182 (4) for the two orientations. The crystal structure is stabilized by a three-dimensional network of C—H···Cl hydrogen bonds.

3D view



Chemical scheme



Structure description

Peralkylated guanidinium ions with complex inorganic anions are considered to be organic–inorganic hybrid compounds. Their physical behaviour makes them interesting for application in scanning electron microscopy (SEM), where the contrast and the brightness of the obtained pictures depend on the heaviest atom present in the anions. By testing various guanidinium salts with different inorganic complex anions, we found out that guanidinium chloridobismuthates and iodidobismuthates are very promising

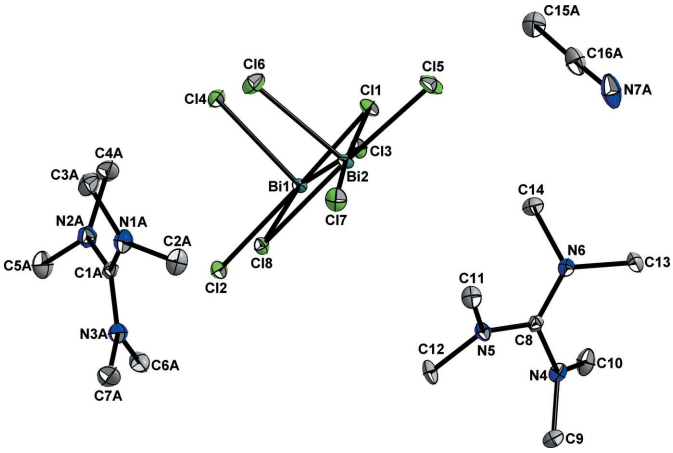


Figure 1
The structure of the solvated title compound with displacement ellipsoids at the 50% probability level. All H atoms have been omitted for clarity. Only the major orientation of the disordered cation I and the acetonitrile molecule is shown.

candidates for this purpose (Knobloch *et al.*, 2016). One of them is the here presented title compound. The asymmetric unit comprises two *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium ions, one half $[Bi_4Cl_{16}]^{4-}$ ion and one acetonitrile molecule (Fig. 1). One cation (cation I) shows orientational disorder and two sets of N and C positions were found, with an occupancy ratio of 0.941 (2):0.059 (2) (Fig. 2). The second cation (cation II) is not disordered. The C—N bond lengths in the two guanidinium ions range from 1.334 (17) to 1.341 (17) Å, indicating partial double-bond character. The N—C—N angles range from 116 (2) to 120.8 (2)°, indicating nearly ideal trigonal-planar surroundings for the carbon centres C1 and C8

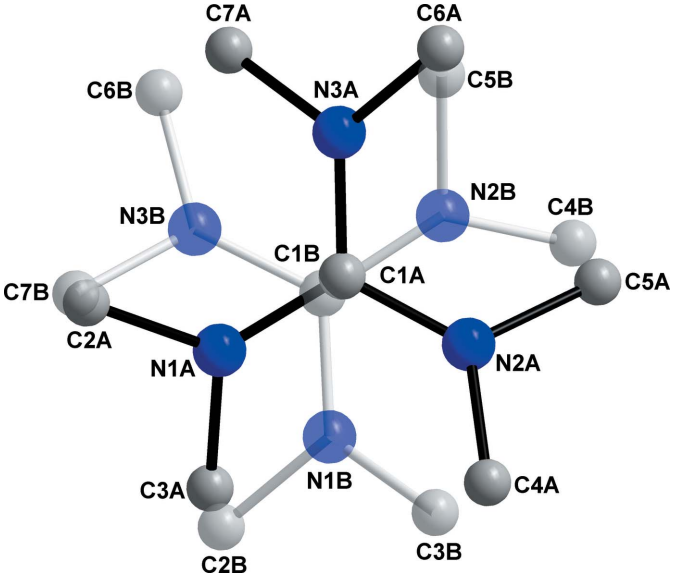


Figure 2
The structure of the orientationally disordered *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium ion (cation I). The N and C atoms are disordered between the opaque and dark positions. All H atoms have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2*B*—H2*E*···Cl2	0.98	2.48	3.382 (3)	153
C3*B*—H3*F*···Cl8^{i}	0.98	2.81	3.464 (3)	125
C4*B*—H4*F*···Cl4	0.98	2.80	3.632 (3)	143
C6*A*—H6*B*···Cl1^{ii}	0.98	2.82	3.577 (3)	134
C6*B*—H6*F*···Cl6^{iii}	0.98	2.67	3.193 (3)	113
C7*B*—H7*E*···Cl5^{ii}	0.98	2.71	3.594 (3)	151
C11—H11*A*···Cl2^{iv}	0.98	2.59	3.488 (3)	153

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

by the nitrogen atoms. The positive charge is completely delocalized on the CN_3 planes. The C—N bond lengths in both cations are in very good agreement with the data from the crystal structure analysis of known *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium salts [see, for example: the tetraphenylborate (Frey *et al.*, 1998), chloride (Oelkers & Sundermeyer, 2011) and cyanate (Tiritiris & Kantlehner, 2015)].

The four Bi^{III} ions are coordinated in a distorted octahedral manner by six chloride ions, with Bi—Cl bond lengths ranging from 2.4982 (7) to 3.0320 (6) Å. Two $[Bi_2Cl_8]^{2-}$ dimers are fused together, forming a centrosymmetric tetranuclear $[Bi_4Cl_{16}]^{4-}$ cluster (Fig. 3). The bond lengths of bismuth to the terminal chlorides [2.4982 (7)–2.5509 (6) Å] are shorter than those of the double and triply bridging ones [2.7052 (6)–3.0320 (6) Å]. The same anionic arrangement was observed in the crystal structure of the compound $[(PPh)_4][Bi_4Cl_{16}]\cdot 3CH_3CN$, where the Bi—Cl bond lengths range from 2.499 (5) to 3.071 (6) Å (Ahmed *et al.*, 2001).

The crystal structure of the title compound is stabilized by C—H···Cl hydrogen bonds, forming a three-dimensional network (Fig. 4, Table 1).

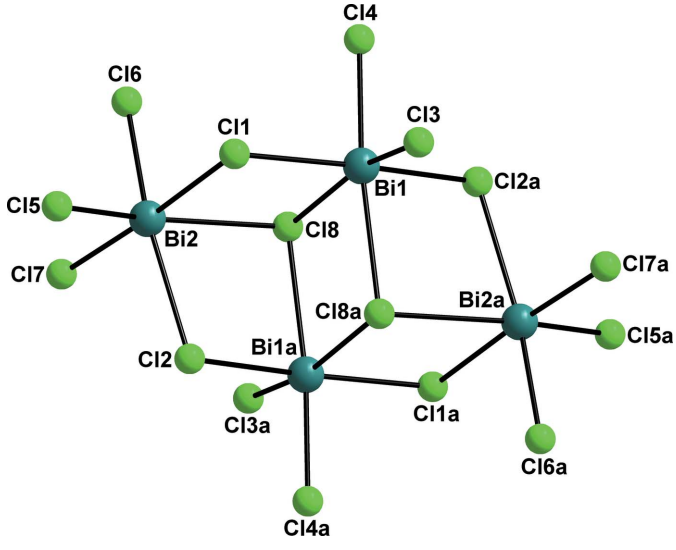


Figure 3
Two $[Bi_2Cl_8]^{2-}$ dimers forming a centrosymmetric tetranuclear $[Bi_4Cl_{16}]^{4-}$ cluster in the structure of the title compound. The other half of the anion is generated by the symmetry operator $(-x, -y+1, -z)$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_7H_{18}N_3)_4[Bi_4Cl_{16}]\cdot 2C_2H_3N$
M_r	2062.20
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	12.6427 (6), 18.8486 (11), 15.1377 (9)
β (°)	112.782 (2)
V (Å^3)	3325.9 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	11.23
Crystal size (mm)	0.20 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker Kappa APEXII DUO
Absorption correction	Multi-scan Blessing, 1995
T_{min}, T_{max}	0.430, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	70365, 10188, 8598
R_{int}	0.047
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.716
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.021, 0.038, 1.01
No. of reflections	10188
No. of parameters	395
No. of restraints	360
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.82, −1.16

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2005).

Synthesis and crystallization

The title compound was obtained by mixing an acetonitrile solution of *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium chloride with $BiCl_3$ dissolved in acetonitrile at room temperature. The colorless precipitate was removed by filtration and it was recrystallized from an acetonitrile solution. After evaporation of the solvent at ambient temperature, colorless single crystals suitable for X-ray analysis emerged.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The atoms C1–C7 and N1–N3 of one cation (cation I) are disordered over two sets of sites (C1*A*/C1*B*–C7*A*/C7*B* and N1*A*/N1*B*–N3*A*/N3*B*) with refined occupancies of 0.941 (2):0.059 (2). The atoms C15, C16 and N7 of the acetonitrile molecule are disordered over two sets of sites (C15*A*/C15*B*, C16*A*/C16*B* and N7*A*/N7*B*) with refined occupancies of 0.818 (4):0.182 (4). The major and minor disordered components were each restrained to have similar geometries. The anisotropic displacement parameters of equivalent guanidinium carbon atoms were constrained to be identical, and the U^{ij} components of all disordered atoms were restrained to be similar if closer than 1.7 Å.

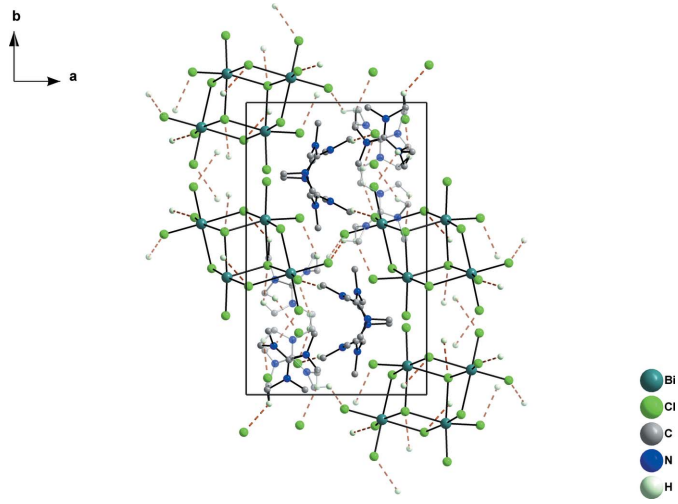


Figure 4
The molecular packing of the title compound (view along *ab*), showing the C—H···Cl hydrogen bonds (brown dashed lines). Both orientations (major: dark; minor: opaque) of the disordered *N*,*N*,*N*′,*N*′,*N*″,*N*″-hexamethylguanidinium ion (cation I) are depicted.

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References

Ahmed, I. A., Blachnik, R. & Reuter, H. (2001). *Z. Anorg. Allg. Chem.* **627**, 2057–2062.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Frey, W., Vettel, M., Edelmann, K. & Kantlehner, W. (1998). *Z. Kristallogr.* **213**, 77–78.
Knobloch, G., Saur, S., Gentner, A. R., Tussetschläger, S., Stein, T., Hader, B. & Kantlehner, W. (2016). *Z. Naturforsch. Teil B*, **71**. Accepted.
Oelkers, B. & Sundermeyer, J. (2011). *Green Chem.* **13**, 608–618.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Tiritiris, I. & Kantlehner, W. (2015). *Acta Cryst.* E**71**, o1076–o1077.

full crystallographic data

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Ioannis Tiritiris, Georg Knobloch, Stefan Saur and Willi Kantlehner

N,*N*,*N*′,*N*′,*N*′′,*N*′′-Hexamethylguanidinium di-μ_3-chlorido-tetra-μ_2-chlorido-decachloridotetrabismuthate acetonitrile disolvate

Crystal data

$(C_7H_{18}N_3)_4[Bi_4Cl_{16}]\cdot 2C_2H_3N$
M_r = 2062.20
Monoclinic, $P2_1/c$
a = 12.6427 (6) Å
b = 18.8486 (11) Å
c = 15.1377 (9) Å
β = 112.782 (2)°
V = 3325.9 (3) Å^3
Z = 2
$F(000)$ = 1944
D_x = 2.059 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 70365 reflections
θ = 1.8–30.6°
μ = 11.23 mm^{-1}
T = 100 K
Block, colorless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker Kappa APEXII DUO diffractometer
Radiation source: fine-focus sealed tube
Triumph monochromator
φ scans, and ω scans
Absorption correction: multi-scan Blessing, 1995
T_{min} = 0.430, T_{max} = 0.746
70365 measured reflections
10188 independent reflections
8598 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 30.6°, θ_{min} = 1.8°
h = −16→18
k = −26→26
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.038
S = 1.01
10188 reflections
395 parameters
360 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0113P)^2 + 0.8846P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −1.16 e Å^{-3}

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Bi1	−0.10623 (2)	0.40160 (2)	−0.01456 (2)	0.00889 (2)	
Bi2	0.24769 (2)	0.41496 (2)	0.19916 (2)	0.01052 (2)	
Cl1	0.01311 (5)	0.37148 (3)	0.17264 (4)	0.01466 (12)	
Cl2	0.28556 (6)	0.39319 (4)	0.37157 (5)	0.02219 (14)	
Cl3	−0.19069 (5)	0.43227 (3)	−0.20501 (4)	0.01665 (13)	
Cl4	0.29154 (6)	0.28873 (3)	0.16379 (5)	0.02239 (14)	
Cl5	−0.29685 (5)	0.39552 (3)	0.00622 (5)	0.01453 (12)	
Cl6	0.45474 (5)	0.45094 (4)	0.24316 (5)	0.02436 (15)	
Cl7	−0.11342 (5)	0.27054 (3)	−0.05307 (5)	0.01789 (13)	
Cl8	0.11940 (5)	0.44264 (3)	−0.01312 (4)	0.01272 (12)	
C1A	0.2366 (2)	0.11935 (15)	0.3368 (3)	0.0121 (5)	0.941 (2)
N1A	0.33230 (18)	0.13654 (12)	0.41131 (17)	0.0159 (5)	0.941 (2)
C2A	0.3957 (5)	0.0846 (3)	0.4848 (2)	0.0194 (9)	0.941 (2)
H2A	0.3502	0.0411	0.4758	0.029*	0.941 (2)
H2B	0.4108	0.1043	0.5484	0.029*	0.941 (2)
H2C	0.4687	0.0736	0.4793	0.029*	0.941 (2)
C3A	0.3775 (3)	0.2088 (2)	0.4288 (3)	0.0208 (8)	0.941 (2)
H3A	0.3390	0.2378	0.3715	0.031*	0.941 (2)
H3B	0.4601	0.2080	0.4438	0.031*	0.941 (2)
H3C	0.3636	0.2292	0.4829	0.031*	0.941 (2)
N2A	0.15245 (19)	0.16682 (11)	0.29975 (16)	0.0153 (5)	0.941 (2)
C4A	0.1233 (3)	0.21768 (17)	0.3592 (2)	0.0208 (7)	0.941 (2)
H4A	0.1672	0.2069	0.4269	0.031*	0.941 (2)
H4B	0.0411	0.2147	0.3455	0.031*	0.941 (2)
H4C	0.1420	0.2657	0.3451	0.031*	0.941 (2)
C5A	0.0805 (3)	0.16864 (19)	0.1982 (2)	0.0222 (7)	0.941 (2)
H5A	0.1161	0.1403	0.1628	0.033*	0.941 (2)
H5B	0.0721	0.2178	0.1754	0.033*	0.941 (2)
H5C	0.0048	0.1491	0.1878	0.033*	0.941 (2)
N3A	0.22668 (19)	0.05542 (11)	0.29608 (16)	0.0155 (5)	0.941 (2)
C6A	0.1168 (5)	0.01769 (17)	0.2567 (4)	0.0219 (8)	0.941 (2)
H6A	0.0606	0.0421	0.2760	0.033*	0.941 (2)
H6B	0.1272	−0.0310	0.2812	0.033*	0.941 (2)
H6C	0.0891	0.0168	0.1866	0.033*	0.941 (2)
C7A	0.3259 (3)	0.01705 (15)	0.2938 (2)	0.0230 (7)	0.941 (2)

H7A	0.3916	0.0494	0.3109	0.035*	0.941 (2)
H7B	0.3080	−0.0017	0.2292	0.035*	0.941 (2)
H7C	0.3448	−0.0223	0.3397	0.035*	0.941 (2)
C1B	0.256 (3)	0.1261 (19)	0.347 (3)	0.0121 (5)	0.059 (2)
N1B	0.257 (2)	0.1880 (13)	0.391 (2)	0.013 (3)	0.059 (2)
C2B	0.366 (3)	0.225 (4)	0.442 (6)	0.0208 (8)	0.059 (2)
H2D	0.4028	0.2054	0.5066	0.031*	0.059 (2)
H2E	0.3522	0.2754	0.4448	0.031*	0.059 (2)
H2F	0.4171	0.2172	0.4071	0.031*	0.059 (2)
C3B	0.157 (3)	0.221 (3)	0.398 (4)	0.0208 (7)	0.059 (2)
H3D	0.1292	0.2592	0.3510	0.031*	0.059 (2)
H3E	0.1766	0.2399	0.4626	0.031*	0.059 (2)
H3F	0.0959	0.1850	0.3850	0.031*	0.059 (2)
N2B	0.161 (2)	0.1065 (15)	0.273 (2)	0.013 (3)	0.059 (2)
C4B	0.103 (5)	0.152 (3)	0.190 (3)	0.0222 (7)	0.059 (2)
H4D	0.0670	0.1921	0.2082	0.033*	0.059 (2)
H4E	0.0446	0.1245	0.1394	0.033*	0.059 (2)
H4F	0.1598	0.1697	0.1655	0.033*	0.059 (2)
C5B	0.124 (8)	0.032 (2)	0.258 (6)	0.0219 (8)	0.059 (2)
H5D	0.0428	0.0288	0.2473	0.033*	0.059 (2)
H5E	0.1697	0.0043	0.3147	0.033*	0.059 (2)
H5F	0.1358	0.0137	0.2019	0.033*	0.059 (2)
N3B	0.336 (2)	0.0776 (14)	0.391 (2)	0.013 (3)	0.059 (2)
C6B	0.376 (4)	0.025 (2)	0.340 (3)	0.0230 (7)	0.059 (2)
H6D	0.3406	0.0339	0.2713	0.035*	0.059 (2)
H6E	0.3557	−0.0227	0.3541	0.035*	0.059 (2)
H6F	0.4599	0.0285	0.3617	0.035*	0.059 (2)
C7B	0.394 (8)	0.075 (5)	0.496 (2)	0.0194 (9)	0.059 (2)
H7D	0.4734	0.0907	0.5146	0.029*	0.059 (2)
H7E	0.3924	0.0263	0.5178	0.029*	0.059 (2)
H7F	0.3540	0.1063	0.5246	0.029*	0.059 (2)
N4	0.67317 (17)	0.24075 (11)	0.16986 (15)	0.0154 (4)	
C8	0.6079 (2)	0.18307 (12)	0.13462 (18)	0.0123 (5)	
N5	0.53427 (17)	0.16194 (11)	0.17368 (15)	0.0142 (4)	
C9	0.6330 (2)	0.30192 (13)	0.2072 (2)	0.0202 (6)	
H9A	0.5504	0.2974	0.1913	0.030*	
H9B	0.6478	0.3454	0.1785	0.030*	
H9C	0.6737	0.3040	0.2770	0.030*	
N6	0.61635 (18)	0.14779 (11)	0.06119 (15)	0.0163 (5)	
C10	0.7919 (2)	0.24393 (14)	0.1767 (2)	0.0225 (6)	
H10A	0.8151	0.1972	0.1622	0.034*	
H10B	0.8425	0.2580	0.2417	0.034*	
H10C	0.7977	0.2788	0.1307	0.034*	
C11	0.4263 (2)	0.12587 (14)	0.11906 (19)	0.0166 (5)	
H11A	0.4094	0.1303	0.0504	0.025*	
H11B	0.3643	0.1476	0.1333	0.025*	
H11C	0.4329	0.0756	0.1368	0.025*	
C12	0.5605 (2)	0.16910 (14)	0.27610 (18)	0.0189 (6)	

H12A	0.6383	0.1881	0.3083	0.028*	
H12B	0.5556	0.1225	0.3030	0.028*	
H12C	0.5053	0.2015	0.2857	0.028*	
C13	0.6450 (2)	0.18217 (14)	−0.01299 (19)	0.0204 (6)	
H13A	0.6432	0.2338	−0.0058	0.031*	
H13B	0.5890	0.1683	−0.0762	0.031*	
H13C	0.7219	0.1675	−0.0067	0.031*	
C14	0.6018 (2)	0.07078 (13)	0.0509 (2)	0.0213 (6)	
H14A	0.5938	0.0511	0.1079	0.032*	
H14B	0.6691	0.0498	0.0437	0.032*	
H14C	0.5329	0.0599	−0.0059	0.032*	
C15A	0.0485 (7)	0.3900 (7)	0.4428 (8)	0.031 (2)	0.818 (4)
H15A	−0.0311	0.3952	0.4368	0.046*	0.818 (4)
H15B	0.0817	0.3471	0.4796	0.046*	0.818 (4)
H15C	0.0504	0.3863	0.3788	0.046*	0.818 (4)
C16A	0.1136 (3)	0.4506 (2)	0.4911 (3)	0.0300 (10)	0.818 (4)
N7A	0.1642 (3)	0.4984 (2)	0.5316 (3)	0.0486 (12)	0.818 (4)
C15B	0.028 (4)	0.389 (3)	0.430 (4)	0.025 (6)	0.182 (4)
H15D	0.0039	0.3927	0.3608	0.038*	0.182 (4)
H15E	0.0436	0.3391	0.4497	0.038*	0.182 (4)
H15F	0.0978	0.4170	0.4621	0.038*	0.182 (4)
C16B	−0.0609 (14)	0.4149 (8)	0.4577 (13)	0.028 (4)	0.182 (4)
N7B	−0.1330 (12)	0.4384 (7)	0.4757 (11)	0.031 (4)	0.182 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Bi1	0.00813 (4)	0.00989 (4)	0.00844 (5)	−0.00030 (3)	0.00299 (3)	0.00070 (3)
Bi2	0.00962 (4)	0.01166 (5)	0.00897 (5)	−0.00038 (3)	0.00217 (3)	0.00118 (3)
Cl1	0.0127 (3)	0.0191 (3)	0.0114 (3)	−0.0001 (2)	0.0038 (2)	0.0026 (2)
Cl2	0.0260 (3)	0.0270 (4)	0.0106 (3)	−0.0074 (3)	0.0038 (3)	0.0024 (3)
Cl3	0.0211 (3)	0.0158 (3)	0.0104 (3)	0.0001 (2)	0.0032 (2)	0.0000 (2)
Cl4	0.0219 (3)	0.0158 (3)	0.0304 (4)	0.0034 (2)	0.0111 (3)	−0.0015 (3)
Cl5	0.0112 (3)	0.0175 (3)	0.0162 (3)	−0.0001 (2)	0.0066 (2)	−0.0009 (2)
Cl6	0.0145 (3)	0.0301 (4)	0.0303 (4)	−0.0076 (3)	0.0107 (3)	−0.0059 (3)
Cl7	0.0217 (3)	0.0123 (3)	0.0219 (4)	−0.0003 (2)	0.0109 (3)	−0.0024 (3)
Cl8	0.0129 (3)	0.0142 (3)	0.0121 (3)	−0.0004 (2)	0.0060 (2)	0.0000 (2)
C1A	0.0115 (13)	0.0105 (12)	0.0168 (14)	−0.0011 (10)	0.0082 (10)	0.0014 (10)
N1A	0.0134 (11)	0.0163 (12)	0.0178 (13)	−0.0009 (9)	0.0060 (9)	0.0025 (10)
C2A	0.0116 (13)	0.023 (2)	0.0214 (17)	0.0007 (14)	0.0040 (13)	0.0068 (14)
C3A	0.0209 (14)	0.018 (2)	0.0250 (19)	−0.0077 (13)	0.0109 (13)	−0.0028 (14)
N2A	0.0165 (11)	0.0156 (12)	0.0146 (12)	0.0032 (9)	0.0069 (10)	0.0026 (9)
C4A	0.0226 (16)	0.0193 (15)	0.0252 (19)	0.0067 (13)	0.0146 (13)	0.0032 (15)
C5A	0.0192 (16)	0.0258 (19)	0.0192 (16)	0.0059 (12)	0.0049 (12)	0.0045 (13)
N3A	0.0171 (11)	0.0136 (11)	0.0192 (13)	−0.0019 (9)	0.0108 (10)	−0.0014 (10)
C6A	0.0256 (15)	0.0218 (18)	0.0206 (16)	−0.0134 (18)	0.0116 (13)	−0.0067 (18)
C7A	0.0273 (17)	0.0192 (15)	0.032 (2)	0.0037 (13)	0.0213 (15)	−0.0004 (14)
C1B	0.0115 (13)	0.0105 (12)	0.0168 (14)	−0.0011 (10)	0.0082 (10)	0.0014 (10)

N1B	0.013 (4)	0.011 (4)	0.017 (4)	0.001 (4)	0.007 (4)	0.000 (4)
C2B	0.0209 (14)	0.018 (2)	0.0250 (19)	−0.0077 (13)	0.0109 (13)	−0.0028 (14)
C3B	0.0226 (16)	0.0193 (15)	0.0252 (19)	0.0067 (13)	0.0146 (13)	0.0032 (15)
N2B	0.014 (4)	0.013 (4)	0.015 (4)	−0.004 (4)	0.009 (4)	0.005 (4)
C4B	0.0192 (16)	0.0258 (19)	0.0192 (16)	0.0059 (12)	0.0049 (12)	0.0045 (13)
C5B	0.0256 (15)	0.0218 (18)	0.0206 (16)	−0.0134 (18)	0.0116 (13)	−0.0067 (18)
N3B	0.013 (4)	0.013 (4)	0.018 (4)	0.001 (4)	0.012 (4)	0.002 (4)
C6B	0.0273 (17)	0.0192 (15)	0.032 (2)	0.0037 (13)	0.0213 (15)	−0.0004 (14)
C7B	0.0116 (13)	0.023 (2)	0.0214 (17)	0.0007 (14)	0.0040 (13)	0.0068 (14)
N4	0.0149 (10)	0.0134 (11)	0.0178 (12)	−0.0010 (8)	0.0063 (9)	0.0012 (9)
C8	0.0135 (12)	0.0107 (12)	0.0123 (13)	0.0018 (9)	0.0046 (10)	0.0025 (10)
N5	0.0138 (10)	0.0174 (11)	0.0110 (11)	−0.0001 (8)	0.0042 (9)	0.0001 (9)
C9	0.0270 (15)	0.0119 (13)	0.0220 (16)	0.0006 (10)	0.0099 (12)	0.0000 (11)
N6	0.0222 (11)	0.0134 (11)	0.0157 (12)	0.0017 (8)	0.0100 (10)	0.0024 (9)
C10	0.0150 (13)	0.0257 (15)	0.0267 (16)	−0.0029 (11)	0.0078 (12)	0.0070 (12)
C11	0.0107 (12)	0.0210 (14)	0.0156 (14)	−0.0031 (10)	0.0025 (10)	−0.0003 (11)
C12	0.0191 (13)	0.0281 (15)	0.0094 (14)	0.0002 (11)	0.0055 (11)	0.0027 (11)
C13	0.0287 (15)	0.0215 (14)	0.0159 (15)	0.0032 (11)	0.0139 (12)	0.0029 (11)
C14	0.0280 (15)	0.0155 (14)	0.0205 (16)	0.0017 (11)	0.0095 (12)	−0.0016 (12)
C15A	0.036 (4)	0.030 (3)	0.028 (4)	0.000 (3)	0.015 (3)	−0.002 (3)
C16A	0.031 (2)	0.040 (2)	0.026 (2)	0.0026 (17)	0.0187 (18)	−0.0040 (18)
N7A	0.044 (2)	0.062 (3)	0.048 (3)	−0.014 (2)	0.027 (2)	−0.026 (2)
C15B	0.033 (10)	0.019 (9)	0.028 (10)	−0.008 (8)	0.015 (9)	−0.008 (8)
C16B	0.041 (8)	0.019 (7)	0.032 (8)	−0.012 (6)	0.021 (6)	−0.008 (6)
N7B	0.040 (8)	0.013 (7)	0.048 (9)	−0.011 (6)	0.027 (7)	−0.014 (6)

Geometric parameters (Å, º)

Bi1—Cl7	2.5317 (6)	N2B—C4B	1.467 (19)
Bi1—Cl5	2.5509 (6)	C4B—H4D	0.9800
Bi1—Cl1	2.7052 (6)	C4B—H4E	0.9800
Bi1—Cl3	2.7206 (6)	C4B—H4F	0.9800
Bi1—Cl8	2.9475 (6)	C5B—H5D	0.9800
Bi1—Cl8^{i}	2.9794 (6)	C5B—H5E	0.9800
Bi2—Cl2	2.4982 (7)	C5B—H5F	0.9800
Bi2—Cl6	2.5309 (6)	N3B—C6B	1.465 (18)
Bi2—Cl4	2.5465 (6)	N3B—C7B	1.465 (19)
Bi2—Cl1	2.9507 (6)	C6B—H6D	0.9800
Bi2—Cl3^{i}	2.9780 (6)	C6B—H6E	0.9800
Bi2—Cl8	3.0320 (6)	C6B—H6F	0.9800
Cl3—Bi2^{i}	2.9780 (6)	C7B—H7D	0.9800
Cl8—Bi1^{i}	2.9794 (6)	C7B—H7E	0.9800
C1A—N1A	1.336 (3)	C7B—H7F	0.9800
C1A—N2A	1.336 (3)	N4—C8	1.344 (3)
C1A—N3A	1.337 (3)	N4—C9	1.459 (3)
N1A—C3A	1.461 (4)	N4—C10	1.466 (3)
N1A—C2A	1.466 (4)	C8—N6	1.335 (3)
C2A—H2A	0.9800	C8—N5	1.342 (3)

C2A—H2B	0.9800	N5—C12	1.461 (3)
C2A—H2C	0.9800	N5—C11	1.461 (3)
C3A—H3A	0.9800	C9—H9A	0.9800
C3A—H3B	0.9800	C9—H9B	0.9800
C3A—H3C	0.9800	C9—H9C	0.9800
N2A—C5A	1.452 (4)	N6—C13	1.458 (3)
N2A—C4A	1.456 (3)	N6—C14	1.464 (3)
C4A—H4A	0.9800	C10—H10A	0.9800
C4A—H4B	0.9800	C10—H10B	0.9800
C4A—H4C	0.9800	C10—H10C	0.9800
C5A—H5A	0.9800	C11—H11A	0.9800
C5A—H5B	0.9800	C11—H11B	0.9800
C5A—H5C	0.9800	C11—H11C	0.9800
N3A—C6A	1.466 (5)	C12—H12A	0.9800
N3A—C7A	1.460 (3)	C12—H12B	0.9800
C6A—H6A	0.9800	C12—H12C	0.9800
C6A—H6B	0.9800	C13—H13A	0.9800
C6A—H6C	0.9800	C13—H13B	0.9800
C7A—H7A	0.9800	C13—H13C	0.9800
C7A—H7B	0.9800	C14—H14A	0.9800
C7A—H7C	0.9800	C14—H14B	0.9800
C1B—N2B	1.334 (17)	C14—H14C	0.9800
C1B—N3B	1.336 (17)	C15A—C16A	1.432 (12)
C1B—N1B	1.341 (17)	C15A—H15A	0.9800
N1B—C3B	1.457 (18)	C15A—H15B	0.9800
N1B—C2B	1.463 (19)	C15A—H15C	0.9800
C2B—H2D	0.9800	C16A—N7A	1.138 (5)
C2B—H2E	0.9800	C15B—C16B	1.43 (2)
C2B—H2F	0.9800	C15B—H15D	0.9800
C3B—H3D	0.9800	C15B—H15E	0.9800
C3B—H3E	0.9800	C15B—H15F	0.9800
C3B—H3F	0.9800	C16B—N7B	1.138 (14)
N2B—C5B	1.46 (2)		

Cl7—Bi1—Cl5	91.899 (19)	H3D—C3B—H3E	109.5
Cl7—Bi1—Cl1	89.55 (2)	N1B—C3B—H3F	109.6
Cl5—Bi1—Cl1	91.813 (19)	H3D—C3B—H3F	109.5
Cl7—Bi1—Cl3	89.94 (2)	H3E—C3B—H3F	109.5
Cl5—Bi1—Cl3	97.95 (2)	C1B—N2B—C5B	121 (3)
Cl1—Bi1—Cl3	170.233 (18)	C1B—N2B—C4B	123 (3)
Cl7—Bi1—Cl8	101.898 (18)	C5B—N2B—C4B	114 (3)
Cl5—Bi1—Cl8	165.626 (18)	N2B—C4B—H4D	109.3
Cl1—Bi1—Cl8	84.364 (17)	N2B—C4B—H4E	109.9
Cl3—Bi1—Cl8	86.198 (18)	H4D—C4B—H4E	109.5
Cl7—Bi1—Cl8^{i}	174.218 (19)	N2B—C4B—H4F	109.2
Cl5—Bi1—Cl8^{i}	85.409 (17)	H4D—C4B—H4F	109.5
Cl1—Bi1—Cl8^{i}	95.641 (18)	H4E—C4B—H4F	109.5
Cl3—Bi1—Cl8^{i}	85.371 (17)	N2B—C5B—H5D	105.9

Cl8—Bi1—Cl8^{i}	81.200 (17)	N2B—C5B—H5E	110.4
Cl2—Bi2—Cl6	90.51 (2)	H5D—C5B—H5E	109.5
Cl2—Bi2—Cl4	95.56 (2)	N2B—C5B—H5F	112.0
Cl6—Bi2—Cl4	91.06 (2)	H5D—C5B—H5F	109.4
Cl2—Bi2—Cl1	82.673 (19)	H5E—C5B—H5F	109.5
Cl6—Bi2—Cl1	173.16 (2)	C1B—N3B—C6B	123 (2)
Cl4—Bi2—Cl1	90.063 (19)	C1B—N3B—C7B	122 (3)
Cl2—Bi2—Cl3^{i}	94.50 (2)	C6B—N3B—C7B	114 (2)
Cl6—Bi2—Cl3^{i}	88.31 (2)	N3B—C6B—H6D	109.4
Cl4—Bi2—Cl3^{i}	169.92 (2)	N3B—C6B—H6E	109.5
Cl1—Bi2—Cl3^{i}	91.750 (17)	H6D—C6B—H6E	109.5
Cl2—Bi2—Cl8	160.554 (19)	N3B—C6B—H6F	109.4
Cl6—Bi2—Cl8	107.88 (2)	H6D—C6B—H6F	109.5
Cl4—Bi2—Cl8	90.49 (2)	H6E—C6B—H6F	109.5
Cl1—Bi2—Cl8	78.850 (16)	N3B—C7B—H7D	109.4
Cl3^{i}—Bi2—Cl8	80.142 (16)	N3B—C7B—H7E	109.4
Bi1—Cl1—Bi2	101.987 (19)	H7D—C7B—H7E	109.5
Bi1—Cl3—Bi2^{i}	100.032 (19)	N3B—C7B—H7F	109.7
Bi1—Cl8—Bi1^{i}	98.801 (17)	H7D—C7B—H7F	109.5
Bi1—Cl8—Bi2	94.683 (17)	H7E—C7B—H7F	109.5
Bi1^{i}—Cl8—Bi2	93.245 (17)	C8—N4—C9	122.7 (2)
N1A—C1A—N2A	120.4 (2)	C8—N4—C10	121.1 (2)
N1A—C1A—N3A	120.0 (2)	C9—N4—C10	116.2 (2)
N2A—C1A—N3A	119.5 (2)	N6—C8—N5	120.8 (2)
C1A—N1A—C3A	122.8 (3)	N6—C8—N4	119.9 (2)
C1A—N1A—C2A	122.0 (3)	N5—C8—N4	119.3 (2)
C3A—N1A—C2A	115.1 (3)	C8—N5—C12	121.9 (2)
N1A—C2A—H2A	109.5	C8—N5—C11	122.9 (2)
N1A—C2A—H2B	109.5	C12—N5—C11	115.0 (2)
H2A—C2A—H2B	109.5	N4—C9—H9A	109.5
N1A—C2A—H2C	109.5	N4—C9—H9B	109.5
H2A—C2A—H2C	109.5	H9A—C9—H9B	109.5
H2B—C2A—H2C	109.5	N4—C9—H9C	109.5
N1A—C3A—H3A	109.5	H9A—C9—H9C	109.5
N1A—C3A—H3B	109.5	H9B—C9—H9C	109.5
H3A—C3A—H3B	109.5	C8—N6—C13	122.9 (2)
N1A—C3A—H3C	109.5	C8—N6—C14	122.4 (2)
H3A—C3A—H3C	109.5	C13—N6—C14	114.6 (2)
H3B—C3A—H3C	109.5	N4—C10—H10A	109.5
C1A—N2A—C5A	122.0 (3)	N4—C10—H10B	109.5
C1A—N2A—C4A	122.2 (3)	H10A—C10—H10B	109.5
C5A—N2A—C4A	115.7 (2)	N4—C10—H10C	109.5
N2A—C4A—H4A	109.5	H10A—C10—H10C	109.5
N2A—C4A—H4B	109.5	H10B—C10—H10C	109.5
H4A—C4A—H4B	109.5	N5—C11—H11A	109.5
N2A—C4A—H4C	109.5	N5—C11—H11B	109.5
H4A—C4A—H4C	109.5	H11A—C11—H11B	109.5
H4B—C4A—H4C	109.5	N5—C11—H11C	109.5

N2A—C5A—H5A	109.5	H11A—C11—H11C	109.5
N2A—C5A—H5B	109.5	H11B—C11—H11C	109.5
H5A—C5A—H5B	109.5	N5—C12—H12A	109.5
N2A—C5A—H5C	109.5	N5—C12—H12B	109.5
H5A—C5A—H5C	109.5	H12A—C12—H12B	109.5
H5B—C5A—H5C	109.5	N5—C12—H12C	109.5
C1A—N3A—C6A	122.0 (3)	H12A—C12—H12C	109.5
C1A—N3A—C7A	122.0 (2)	H12B—C12—H12C	109.5
C6A—N3A—C7A	116.0 (3)	N6—C13—H13A	109.5
N3A—C6A—H6A	109.6	N6—C13—H13B	109.5
N3A—C6A—H6B	109.4	H13A—C13—H13B	109.5
H6A—C6A—H6B	109.5	N6—C13—H13C	109.5
N3A—C6A—H6C	109.4	H13A—C13—H13C	109.5
H6A—C6A—H6C	109.5	H13B—C13—H13C	109.5
H6B—C6A—H6C	109.5	N6—C14—H14A	109.5
N3A—C7A—H7A	109.5	N6—C14—H14B	109.5
N3A—C7A—H7B	109.5	H14A—C14—H14B	109.5
H7A—C7A—H7B	109.5	N6—C14—H14C	109.5
N3A—C7A—H7C	109.5	H14A—C14—H14C	109.5
H7A—C7A—H7C	109.5	H14B—C14—H14C	109.5
H7B—C7A—H7C	109.5	C16A—C15A—H15A	109.5
N2B—C1B—N3B	120 (2)	C16A—C15A—H15B	109.5
N2B—C1B—N1B	120 (2)	H15A—C15A—H15B	109.5
N3B—C1B—N1B	119 (2)	C16A—C15A—H15C	109.5
C1B—N1B—C3B	124 (2)	H15A—C15A—H15C	109.5
C1B—N1B—C2B	120 (2)	H15B—C15A—H15C	109.5
C3B—N1B—C2B	116 (2)	N7A—C16A—C15A	178.0 (6)
N1B—C2B—H2D	109.4	C16B—C15B—H15D	109.5
N1B—C2B—H2E	109.6	C16B—C15B—H15E	109.5
H2D—C2B—H2E	109.5	H15D—C15B—H15E	109.5
N1B—C2B—H2F	109.4	C16B—C15B—H15F	109.5
H2D—C2B—H2F	109.5	H15D—C15B—H15F	109.5
H2E—C2B—H2F	109.5	H15E—C15B—H15F	109.5
N1B—C3B—H3D	109.4	N7B—C16B—C15B	176 (3)
N1B—C3B—H3E	109.3		

N2A—C1A—N1A—C3A	−29.6 (5)	N3B—C1B—N2B—C4B	−144 (5)
N3A—C1A—N1A—C3A	147.4 (3)	N1B—C1B—N2B—C4B	53 (8)
N2A—C1A—N1A—C2A	146.6 (4)	N2B—C1B—N3B—C6B	45 (8)
N3A—C1A—N1A—C2A	−36.4 (5)	N1B—C1B—N3B—C6B	−152 (5)
N1A—C1A—N2A—C5A	146.7 (3)	N2B—C1B—N3B—C7B	−137 (7)
N3A—C1A—N2A—C5A	−30.3 (5)	N1B—C1B—N3B—C7B	26 (9)
N1A—C1A—N2A—C4A	−36.4 (5)	C9—N4—C8—N6	145.2 (2)
N3A—C1A—N2A—C4A	146.5 (3)	C10—N4—C8—N6	−37.6 (3)
N1A—C1A—N3A—C6A	145.0 (4)	C9—N4—C8—N5	−34.4 (3)
N2A—C1A—N3A—C6A	−37.9 (5)	C10—N4—C8—N5	142.8 (2)
N1A—C1A—N3A—C7A	−31.0 (5)	N6—C8—N5—C12	145.5 (2)
N2A—C1A—N3A—C7A	146.1 (3)	N4—C8—N5—C12	−34.9 (3)

N2B—C1B—N1B—C3B	30 (8)	N6—C8—N5—C11	−30.2 (4)
N3B—C1B—N1B—C3B	−133 (5)	N4—C8—N5—C11	149.5 (2)
N2B—C1B—N1B—C2B	−155 (6)	N5—C8—N6—C13	148.4 (2)
N3B—C1B—N1B—C2B	42 (9)	N4—C8—N6—C13	−31.2 (4)
N3B—C1B—N2B—C5B	19 (10)	N5—C8—N6—C14	−34.0 (4)
N1B—C1B—N2B—C5B	−144 (7)	N4—C8—N6—C14	146.4 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2*B*—H2*E*···Cl2	0.98	2.48	3.382 (3)	153
C3*B*—H3*F*···Cl8^{ii}	0.98	2.81	3.464 (3)	125
C4*B*—H4*F*···Cl4	0.98	2.80	3.632 (3)	143
C6*A*—H6*B*···Cl1^{iii}	0.98	2.82	3.577 (3)	134
C6*B*—H6*F*···Cl6^{iv}	0.98	2.67	3.193 (3)	113
C7*B*—H7*E*···Cl5^{iii}	0.98	2.71	3.594 (3)	151
C11—H11*A*···Cl2^{v}	0.98	2.59	3.488 (3)	153

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