

Bis(3-azaniumylpropyl)azanium hexachloridobismuthate(III) monohydrate

Nizar Elfaleh, Hassen Chouaib* and Slaheddine Kamoun

Laboratoire de Génie des Matériaux et Environnement (LR11ES46), BP 1173, ENIS, Sfax, Tunisia

Correspondence e-mail: chouaib.hassen@yahoo.fr

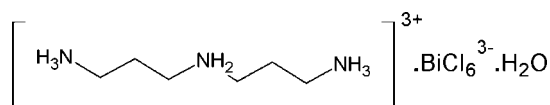
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(C-C) = 0.005$ Å; R factor = 0.021; wR factor = 0.057; data-to-parameter ratio = 31.7.

The asymmetric unit of the title compound, $(C_6H_{20}N_3)\text{-}[\text{BiCl}_6]\cdot\text{H}_2\text{O}$, consists of a triprotonated bis(3-azaniumylpropyl)azanium cation, two halves of an octahedral $[\text{BiCl}_6]^{3-}$ anion, each of the Bi^{III} atoms lying on an inversion centre, and a water molecule. In the crystal, the anions and water molecules are linked by $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming chains running parallel to $[0\bar{1}1]$. The anionic chains and the cations are further linked into a three-dimensional network by $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond interactions.

Related literature

For related structures, see: Chaabouni *et al.* (1998); Fu *et al.* (2005); Rhandour *et al.* (2011); Ouasri *et al.* (2013). For bond-valence-sum calculations, see: Brown & Altermatt (1985). For van der Waals radii, see: Pauling (1960).



Experimental

Crystal data

$(C_6H_{20}N_3)[\text{BiCl}_6]\cdot\text{H}_2\text{O}$

$M_r = 573.95$

Triclinic, $P\bar{1}$

$a = 7.6891$ (1) Å

$b = 10.8642$ (1) Å

$c = 11.9867$ (1) Å

$\alpha = 93.349$ (1)°

$\beta = 108.509$ (1)°

$\gamma = 109.387$ (1)°

$V = 880.54$ (2) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 10.91$ mm⁻¹

$T = 296$ K

$0.1 \times 0.1 \times 0.1$ mm

Data collection

Bruker APEXII CCD diffractometer

Absorption correction: multi-scan

(*SADABS*; Bruker, 2006)

$T_{\text{min}} = 0.336$, $T_{\text{max}} = 0.349$

11734 measured reflections

5325 independent reflections

4009 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$

$wR(F^2) = 0.057$

$S = 0.92$

5325 reflections

168 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 1.07$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.90$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl4}^{\text{i}}$	0.89	2.34	3.174 (3)	156
$\text{N1}-\text{H1B}\cdots\text{Cl2}^{\text{ii}}$	0.89	2.73	3.339 (3)	127
$\text{N1}-\text{H1C}\cdots\text{Cl3}^{\text{iii}}$	0.89	2.82	3.474 (3)	132
$\text{N1}-\text{H1C}\cdots\text{Cl3}^{\text{iv}}$	0.89	2.43	3.293 (3)	163
$\text{N2}-\text{H2B}\cdots\text{OW}$	0.90	1.91	2.804 (4)	173
$\text{N2}-\text{H2A}\cdots\text{Cl2}^{\text{iii}}$	0.90	2.63	3.316 (3)	134
$\text{N2}-\text{H2A}\cdots\text{Cl1}^{\text{ii}}$	0.90	2.71	3.347 (3)	129
$\text{N3}-\text{H3A}\cdots\text{Cl6}$	0.89	2.48	3.362 (3)	169
$\text{N3}-\text{H3B}\cdots\text{Cl1}^{\text{iv}}$	0.89	2.81	3.412 (3)	126
$\text{N3}-\text{H3B}\cdots\text{Cl3}^{\text{v}}$	0.89	2.81	3.612 (3)	151
$\text{N3}-\text{H3C}\cdots\text{Cl5}^{\text{vi}}$	0.89	2.44	3.269 (3)	155
$\text{OW}-\text{H1W}\cdots\text{Cl6}$	0.84 (2)	2.83 (3)	3.620 (4)	157 (6)
$\text{OW}-\text{H2W}\cdots\text{Cl3}^{\text{ii}}$	0.86 (2)	2.82 (4)	3.478 (4)	135 (5)

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINTE* (Bruker, 2006); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ5091).

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supporting information

Acta Cryst. (2013). E69, m666 [doi:10.1107/S1600536813030900]

Bis(3-azaniumylpropyl)azanium hexachloridobismuthate(III) monohydrate

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S1. Comment

This work is a part of our study on the crystal structure of alkylpolyammoniumbismuthate(III) chlorides. This investigation was extended to aliphatic diamines of general formula $\text{NH}_2(\text{CH}_2)_n\text{NH}_2$ (Chaabouni *et al.*, 1998; Rhandour *et al.*, 2011; Ouasri *et al.*, 2013) and triamines of general formula $\text{NH}_2(\text{CH}_2)_n\text{NH}(\text{CH}_2)_n\text{NH}_2$ (Fu *et al.*, 2005) in order to examine the effect of the flexible cation on the bismuth(III) coordination geometry. In these compounds the Bi atom shows a tendency toward distorted octahedral coordination with some rather long Bi—Cl bonds, which is attributed to the aspherical distribution of the lone pair electrons at Bi(III).

The asymmetric unit of the title compound contains one fully protonated bis(3-azaniumylpropyl)azanium cation, two half of a $[\text{BiCl}_6]^{3-}$ anion and a neutral water molecule. A perspective view of the arrangement of these constituent entities is shown in Fig. 1 together with the atom numbering scheme. Two slightly distorted $[\text{BiCl}_6]^{3-}$ octahedra are located in special position on an inversion centre. The Bi—Cl bond lengths vary from 2.6817 (8) to 2.7209 (8) Å with an average bond lengths of 2.7014 (8) Å. These values are much shorter than the sum of the van der Waal radii of Bi and Cl (4.7 Å) according to Pauling (Pauling, 1960). In addition to the bond length differences, the Cl—Bi—Cl angles for the Cl atoms in *cis* position with respect to each other fall in the range of 85.80 (3)–94.20 (3)°. It should be mentioned that the Cl—Bi—Cl bond angles deviate substantially from 90° by 4.2° for Bi(1) and 3.1° for Bi(2). By taking into account the sixth-fold coordination of bismuth atoms, we have proceeded to calculate the bond-valence sum (BVS) of this metal using the parameters given by Brown (Brown *et al.*, 1985). The BVS calculation of the Bi1 and Bi2 ions gave respectively values of 3.23 and 3.38 valence units. These results confirm the presumed oxidation state of Bi(III). The distortion of the $[\text{BiCl}_6]^{3-}$ octahedral are correlated primary to the deformations resulting from the stereochemical activity of the Bi lone electron pair and secondary to deformations resulting from hydrogen bonding interactions. The $[\text{BiCl}_6]^{3-}$ anions are connected through O—H \cdots Cl hydrogen bonds (Table 1), so that $[\text{BiCl}_6(\text{H}_2\text{O})]_n^{3n-}$ chains spread one-dimensionally parallel to the [0 -1 1] direction. The unit cell is crossed by two centrosymmetrical $[\text{BiCl}_6(\text{H}_2\text{O})]_n^{3n-}$ chains with the (0 2 2) mid plane as shown in Fig. 2. The total negative charge (-3) on the framework is balanced by the presence of one independent fully protonated $[\text{NH}_3(\text{CH}_2)_3\text{NH}_2(\text{CH}_2)_3\text{NH}_3]^{3+}$ cation. The major contributions to the cohesion and the stability of the structure is provided by the presence of N—H \cdots Cl and N—H \cdots O hydrogen bonds linkages between the cations and the anionic chains belonging to adjacent (0 2 2) planes. All of these hydrogen bonds, N—H \cdots Cl, N—H \cdots O and O—H \cdots Cl, give rise to a three-dimensional network in the structure (Fig. 3 and Table 1).

S2. Experimental

Crystals of the title compound were obtained by dissolving in 100 ml of a solution of HCl (12M) a stoichiometric mixture of bismuth(III) oxide and bis(3-amino-propyl)amine (molar ratio 1:2). The resulting aqueous solution was then kept at room temperature. After several weeks of slow evaporation at room temperature, prismatic shaped monocrystals of the title compound were obtained. They were washed with diethyl ether and dried for 4 h over CaCl_2 .

S3. Refinement

All H atoms belonging to the organic group cation were geometrically positioned and treated as riding on their parent atoms, with C—H = 0.97 Å and N—H = 0.89–0.90 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$. The water H atoms were located in a difference Fourier map and refined using DFIX and DANG restraints. Their bond lengths were set to ideal values of 0.85 Å and finally they were refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

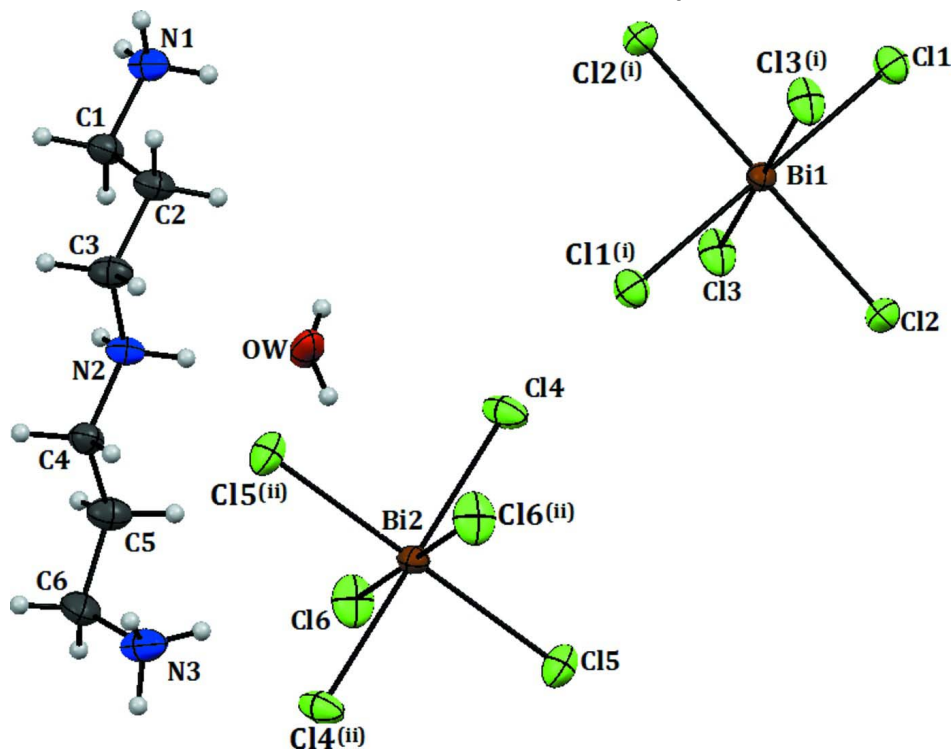


Figure 1

A view of the asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+1, -z$.

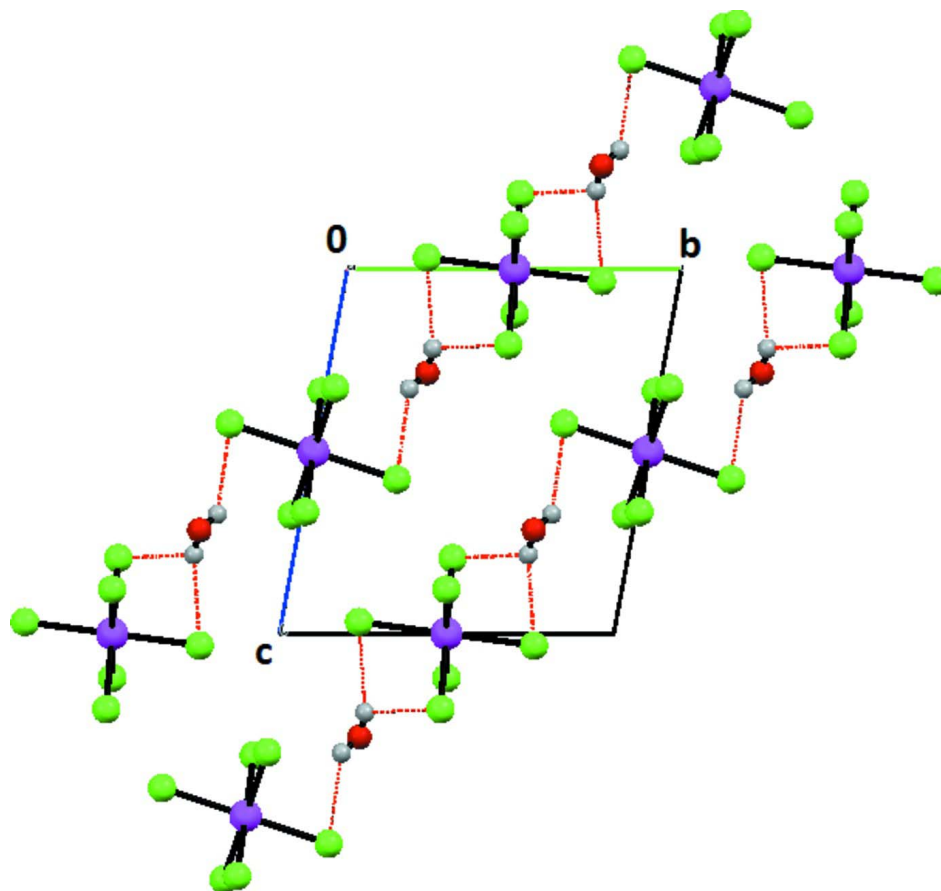


Figure 2

Arrangement of the anionic chains viewed along the a axis (purple= bismuth, green = chloride, red = oxygen, grey = hydrogen). Intermolecular hydrogen bonding is shown as red dashed lines.

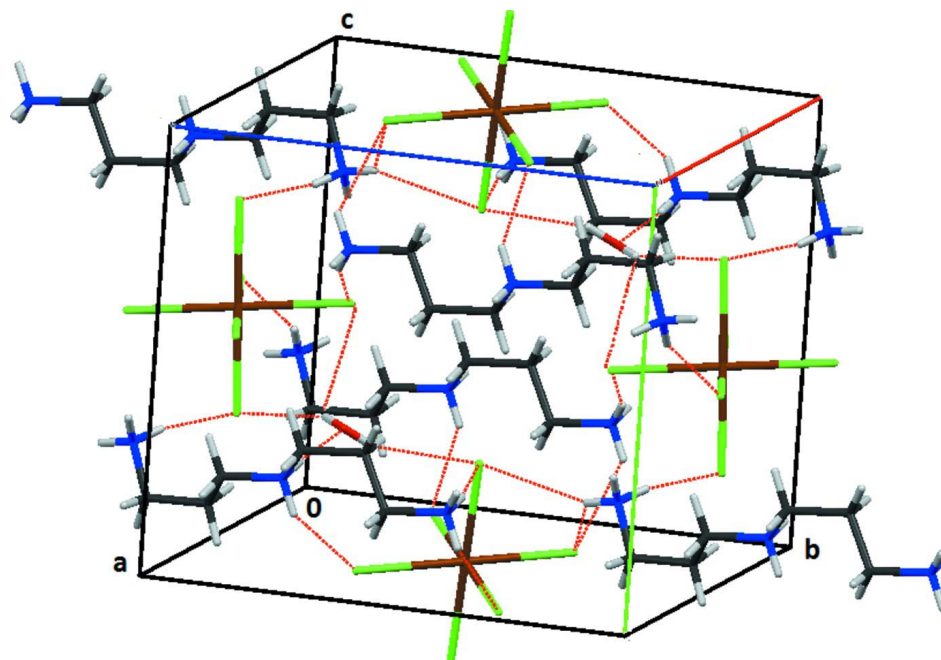


Figure 3

Crystal packing of the title compound showing the hydrogen bonding network as red dashed lines.

Bis(3-azaniumylpropyl)azanium hexachloridobismuthate(III) monohydrate

Crystal data

$(C_6H_{20}N_3)[BiCl_6] \cdot H_2O$

$M_r = 573.95$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.6891(1) \text{ \AA}$

$b = 10.8642(1) \text{ \AA}$

$c = 11.9867(1) \text{ \AA}$

$\alpha = 93.349(1)^\circ$

$\beta = 108.509(1)^\circ$

$\gamma = 109.387(1)^\circ$

$V = 880.54(2) \text{ \AA}^3$

$Z = 2$

$F(000) = 544$

$Z = 2$

$D_x = 2.165 \text{ Mg m}^{-3}$

$D_m = 2.160 \text{ Mg m}^{-3}$

D_m measured by Flotation

Melting point: 430 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

$\theta = 1.8\text{--}30.6^\circ$

$\mu = 10.91 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism, white

$0.1 \times 0.1 \times 0.1 \text{ mm}$

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2006)

$T_{\min} = 0.336$, $T_{\max} = 0.349$

11734 measured reflections

5325 independent reflections

4009 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 30.6^\circ$, $\theta_{\min} = 1.8^\circ$

$h = -10 \rightarrow 10$

$k = -13 \rightarrow 15$

$l = -16 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.021$

$wR(F^2) = 0.057$

$S = 0.92$

5325 reflections

168 parameters

3 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.07 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.90 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0132 (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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Bi1	0.5000	1.0000	0.5000	0.02401 (5)
Bi2	0.5000	0.5000	0.0000	0.02614 (6)
Cl1	0.32827 (12)	1.00845 (8)	0.66539 (8)	0.03793 (18)
Cl2	0.19824 (12)	1.02948 (9)	0.32564 (8)	0.04104 (19)
Cl3	0.31297 (13)	0.73550 (8)	0.42359 (10)	0.0451 (2)
Cl4	0.43949 (12)	0.52414 (10)	0.20718 (8)	0.0461 (2)
Cl5	0.12577 (12)	0.47555 (9)	-0.12177 (9)	0.0468 (2)
Cl6	0.36405 (14)	0.23214 (9)	-0.03209 (11)	0.0523 (3)
N1	1.1331 (4)	0.2550 (3)	0.6613 (2)	0.0383 (6)
H1A	1.2346	0.3229	0.7126	0.057*
H1B	1.1303	0.1800	0.6881	0.057*
H1C	1.0211	0.2667	0.6541	0.057*
C1	1.1551 (5)	0.2473 (3)	0.5432 (3)	0.0351 (7)
H1E	1.0550	0.1664	0.4906	0.042*
H1D	1.2834	0.2440	0.5526	0.042*
C2	1.1357 (5)	0.3649 (3)	0.4873 (3)	0.0368 (7)
H2E	1.2356	0.4457	0.5403	0.044*
H2D	1.0074	0.3681	0.4782	0.044*
C3	1.1583 (5)	0.3592 (3)	0.3652 (3)	0.0378 (8)
H3E	1.1698	0.4437	0.3396	0.045*
H3D	1.2784	0.3441	0.3720	0.045*
N2	0.9871 (4)	0.2518 (2)	0.2741 (2)	0.0316 (6)
H2A	0.9784	0.1738	0.2983	0.038*

H2B	0.8761	0.2653	0.2702	0.038*
C4	0.9967 (5)	0.2418 (3)	0.1523 (3)	0.0328 (7)
H4E	1.1114	0.2220	0.1539	0.039*
H4D	1.0091	0.3258	0.1254	0.039*
C5	0.8107 (5)	0.1329 (3)	0.0668 (3)	0.0408 (8)
H5E	0.6966	0.1481	0.0734	0.049*
H5D	0.8065	0.0483	0.0903	0.049*
C6	0.7976 (5)	0.1254 (4)	-0.0631 (3)	0.0472 (9)
H6E	0.9246	0.1324	-0.0670	0.057*
H6D	0.7013	0.0398	-0.1090	0.057*
N3	0.7405 (4)	0.2322 (3)	-0.1174 (3)	0.0479 (8)
H3A	0.6490	0.2444	-0.0925	0.072*
H3B	0.6922	0.2089	-0.1968	0.072*
H3C	0.8461	0.3071	-0.0955	0.072*
OW	0.6532 (5)	0.2968 (4)	0.2841 (4)	0.0799 (11)
H1W	0.576 (7)	0.300 (7)	0.217 (3)	0.15 (3)*
H2W	0.597 (8)	0.252 (6)	0.328 (5)	0.16 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Bi1	0.02304 (8)	0.02366 (8)	0.02407 (9)	0.00824 (5)	0.00767 (6)	0.00183 (6)
Bi2	0.02630 (8)	0.02556 (9)	0.02270 (9)	0.00658 (6)	0.00748 (6)	0.00122 (6)
Cl1	0.0370 (4)	0.0396 (4)	0.0405 (5)	0.0124 (3)	0.0195 (4)	0.0091 (3)
Cl2	0.0443 (4)	0.0527 (5)	0.0313 (4)	0.0292 (4)	0.0085 (3)	0.0076 (4)
Cl3	0.0399 (4)	0.0281 (4)	0.0656 (6)	0.0074 (3)	0.0238 (4)	-0.0002 (4)
Cl4	0.0397 (4)	0.0576 (5)	0.0310 (4)	0.0018 (4)	0.0186 (4)	-0.0010 (4)
Cl5	0.0317 (4)	0.0527 (5)	0.0532 (6)	0.0183 (4)	0.0086 (4)	0.0101 (4)
Cl6	0.0520 (5)	0.0267 (4)	0.0794 (8)	0.0109 (3)	0.0287 (5)	0.0106 (4)
N1	0.0349 (13)	0.0429 (16)	0.0286 (15)	0.0058 (11)	0.0097 (12)	0.0053 (12)
C1	0.0452 (17)	0.0347 (17)	0.0255 (16)	0.0160 (14)	0.0120 (14)	0.0030 (13)
C2	0.0467 (18)	0.0337 (17)	0.0257 (17)	0.0122 (14)	0.0109 (15)	0.0010 (13)
C3	0.0391 (16)	0.0335 (17)	0.0292 (18)	0.0019 (13)	0.0100 (14)	0.0037 (13)
N2	0.0374 (14)	0.0322 (15)	0.0219 (14)	0.0101 (11)	0.0094 (12)	0.0054 (11)
C4	0.0374 (15)	0.0355 (17)	0.0258 (16)	0.0138 (13)	0.0115 (13)	0.0053 (13)
C5	0.0507 (19)	0.0321 (17)	0.0313 (19)	0.0098 (14)	0.0104 (16)	0.0031 (14)
C6	0.0480 (19)	0.052 (2)	0.035 (2)	0.0197 (17)	0.0085 (17)	-0.0107 (17)
N3	0.0423 (16)	0.059 (2)	0.0293 (16)	0.0076 (14)	0.0075 (13)	0.0059 (14)
OW	0.089 (2)	0.114 (3)	0.088 (3)	0.069 (2)	0.055 (2)	0.067 (2)

Geometric parameters (Å, °)

Bi1—Cl3	2.6976 (8)	C2—H2D	0.9700
Bi1—Cl3 ⁱ	2.6976 (8)	C3—N2	1.485 (4)
Bi1—Cl2	2.7105 (8)	C3—H3E	0.9700
Bi1—Cl2 ⁱ	2.7105 (8)	C3—H3D	0.9700
Bi1—Cl1 ⁱ	2.7209 (8)	N2—C4	1.485 (4)
Bi1—Cl1	2.7209 (8)	N2—H2A	0.9000

Bi2—C14 ⁱⁱ	2.6816 (8)	N2—H2B	0.9000
Bi2—C14	2.6817 (8)	C4—C5	1.517 (4)
Bi2—C15	2.6948 (8)	C4—H4E	0.9700
Bi2—C15 ⁱⁱ	2.6948 (8)	C4—H4D	0.9700
Bi2—C16	2.7025 (9)	C5—C6	1.524 (5)
Bi2—C16 ⁱⁱ	2.7025 (9)	C5—H5E	0.9700
N1—C1	1.478 (4)	C5—H5D	0.9700
N1—H1A	0.8900	C6—N3	1.485 (5)
N1—H1B	0.8900	C6—H6E	0.9700
N1—H1C	0.8900	C6—H6D	0.9700
C1—C2	1.506 (5)	N3—H3A	0.8900
C1—H1E	0.9700	N3—H3B	0.8900
C1—H1D	0.9700	N3—H3C	0.8900
C2—C3	1.528 (5)	OW—H1W	0.844 (19)
C2—H2E	0.9700	OW—H2W	0.857 (19)
C13—Bi1—C13 ⁱ	180.0	C1—C2—H2E	109.1
C13—Bi1—C12	87.59 (3)	C3—C2—H2E	109.1
C13 ⁱ —Bi1—C12	92.41 (3)	C1—C2—H2D	109.1
C13—Bi1—C12 ⁱ	92.41 (3)	C3—C2—H2D	109.1
C13 ⁱ —Bi1—C12 ⁱ	87.59 (3)	H2E—C2—H2D	107.9
C12—Bi1—C12 ⁱ	180.0	N2—C3—C2	111.4 (3)
C13—Bi1—C11 ⁱ	85.80 (3)	N2—C3—H3E	109.4
C13 ⁱ —Bi1—C11 ⁱ	94.20 (3)	C2—C3—H3E	109.4
C12—Bi1—C11 ⁱ	87.82 (3)	N2—C3—H3D	109.4
C12 ⁱ —Bi1—C11 ⁱ	92.18 (3)	C2—C3—H3D	109.4
C13—Bi1—C11	94.20 (3)	H3E—C3—H3D	108.0
C13 ⁱ —Bi1—C11	85.80 (3)	C4—N2—C3	114.5 (3)
C12—Bi1—C11	92.18 (3)	C4—N2—H2A	108.6
C12 ⁱ —Bi1—C11	87.82 (3)	C3—N2—H2A	108.6
C11 ⁱ —Bi1—C11	180.0	C4—N2—H2B	108.6
C14 ⁱⁱ —Bi2—C14	180.00 (4)	C3—N2—H2B	108.6
C14 ⁱⁱ —Bi2—C15	89.74 (3)	H2A—N2—H2B	107.6
C14—Bi2—C15	90.26 (3)	N2—C4—C5	109.4 (3)
C14 ⁱⁱ —Bi2—C15 ⁱⁱ	90.26 (3)	N2—C4—H4E	109.8
C14—Bi2—C15 ⁱⁱ	89.74 (3)	C5—C4—H4E	109.8
C15—Bi2—C15 ⁱⁱ	180.00 (6)	N2—C4—H4D	109.8
C14 ⁱⁱ —Bi2—C16	86.87 (3)	C5—C4—H4D	109.8
C14—Bi2—C16	93.13 (3)	H4E—C4—H4D	108.2
C15—Bi2—C16	87.00 (3)	C4—C5—C6	113.0 (3)
C15 ⁱⁱ —Bi2—C16	93.00 (3)	C4—C5—H5E	109.0
C14 ⁱⁱ —Bi2—C16 ⁱⁱ	93.13 (3)	C6—C5—H5E	109.0
C14—Bi2—C16 ⁱⁱ	86.87 (3)	C4—C5—H5D	109.0
C15—Bi2—C16 ⁱⁱ	93.00 (3)	C6—C5—H5D	109.0
C15 ⁱⁱ —Bi2—C16 ⁱⁱ	87.00 (3)	H5E—C5—H5D	107.8
C16—Bi2—C16 ⁱⁱ	180.00 (6)	N3—C6—C5	112.2 (3)
C1—N1—H1A	109.5	N3—C6—H6E	109.2
C1—N1—H1B	109.5	C5—C6—H6E	109.2

H1A—N1—H1B	109.5	N3—C6—H6D	109.2
C1—N1—H1C	109.5	C5—C6—H6D	109.2
H1A—N1—H1C	109.5	H6E—C6—H6D	107.9
H1B—N1—H1C	109.5	C6—N3—H3A	109.5
N1—C1—C2	111.4 (3)	C6—N3—H3B	109.5
N1—C1—H1E	109.4	H3A—N3—H3B	109.5
C2—C1—H1E	109.4	C6—N3—H3C	109.5
N1—C1—H1D	109.4	H3A—N3—H3C	109.5
C2—C1—H1D	109.4	H3B—N3—H3C	109.5
H1E—C1—H1D	108.0	H1W—OW—H2W	115 (3)
C1—C2—C3	112.3 (3)		
N1—C1—C2—C3	179.9 (3)	C3—N2—C4—C5	-177.7 (3)
C1—C2—C3—N2	69.7 (4)	N2—C4—C5—C6	173.8 (3)
C2—C3—N2—C4	178.8 (3)	C4—C5—C6—N3	-76.5 (4)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots C14 ⁱⁱⁱ	0.89	2.34	3.174 (3)	156
N1—H1B \cdots C12 ^{iv}	0.89	2.73	3.339 (3)	127
N1—H1B \cdots C11 ^v	0.89	2.82	3.474 (3)	132
N1—H1C \cdots C13 ^{iv}	0.89	2.43	3.293 (3)	163
N2—H2B \cdots OW	0.90	1.91	2.804 (4)	173
N2—H2A \cdots C12 ^v	0.90	2.63	3.316 (3)	134
N2—H2A \cdots C11 ^{iv}	0.90	2.71	3.347 (3)	129
N3—H3A \cdots C16	0.89	2.48	3.362 (3)	169
N3—H3B \cdots C11 ^{vi}	0.89	2.81	3.412 (3)	126
N3—H3B \cdots C13 ⁱⁱ	0.89	2.81	3.612 (3)	151
N3—H3C \cdots C15 ^{vii}	0.89	2.44	3.269 (3)	155
OW—H1W \cdots C16	0.84 (2)	2.83 (3)	3.620 (4)	157 (6)
OW—H2W \cdots C13 ^{iv}	0.86 (2)	2.82 (4)	3.478 (4)	135 (5)

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