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Tris(pyrrolidin-1-yl)carbenium tri-μ-iodido-bis[tri-iodidobismuthate(III)]

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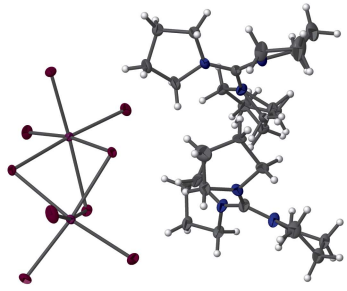
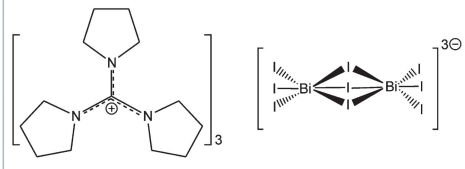
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $3(C_{13}H_{24}N_3)^+ [Bi_2I_9]^{3-}$, comprises two cations and one half of a $[Bi_2I_9]^{3-}$ ion. The C—N bond lengths of the CN_3 units in both cations range from 1.336 (3) to 1.364 (5) Å, indicating partial double-bond character pointing towards charge delocalization within the NCN planes. All five-membered rings adopt an envelope conformation with the C atoms as the flap. One of the pyrrolidine rings (cation I) is disordered over two alternative envelope conformations. Two sets of positions were found for two of the methylene groups with an occupancy ratio of 0.757 (10):0.243 (10). The second disordered pyrrolidine moiety (cation II) is disordered around a twofold rotation axis and exhibits two half-occupied symmetry equivalent counterparts. The two Bi^{III} ions are coordinated by six iodide ions in a distorted octahedral manner, with the Bi–I bond lengths ranging from 2.9544 (2) to 3.2414 (2) Å. Two $[BiI_6]^{3-}$ octahedra are fused together through face-sharing, forming a dinuclear $[Bi_2I_9]^{3-}$ unit. The bond lengths of bismuth to the terminal iodides [2.9544 (2)–2.9889 (2) Å] are shorter than the bridging ones [3.1450 (2)–3.2414 (2) Å].

3D view



Chemical scheme



Structure description

Peralkylated guanidinium ions with complex inorganic anions are considered as 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 iodobismuthates(III) are very suitable candidates for this purpose (Knobloch *et al.*, 2016). One of them is the here presented title compound.

The asymmetric unit comprises two tris(pyrrolidin-1-yl)carbenium ions and one half of a $[Bi_2I_9]^{3-}$ ion (Fig. 1). All five-membered rings adopt an envelope conformation with the

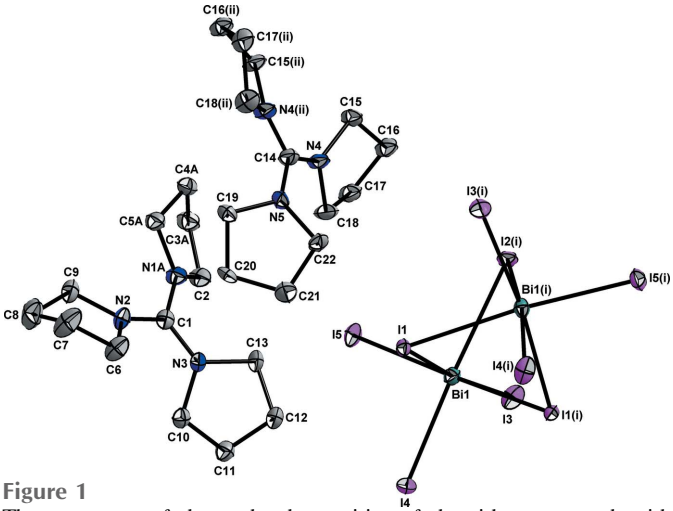


Figure 1
The structures of the molecular entities of the title compound, with displacement ellipsoids at the 50% probability level. All H atoms have been omitted for clarity. Only one moiety of the disordered pyrrolidine ring of cation II and the major orientation of the disordered pyrrolidine ring of cation I are shown [symmetry codes: (i) $1-x, y, \frac{1}{2}-z$; (ii) $-x, y, \frac{1}{2}-z$].

C atoms as the flap. One of the pyrrolidine rings (cation I) is disordered over two alternative envelope conformations (details of the disorder are described in *Refinement*). The second pyrrolidine moiety (cation II) is also disordered (Fig. 2). The C—N bond lengths of the CN_3 units in both cations range from 1.336 (3) to 1.364 (5) Å, indicating partial double-bond character. The N—C—N angles range from 119.4 (2) to 121.7 (9)°, indicating nearly ideal trigonal–planar surroundings of the carbon atoms C1 and C14 by the nitrogen atoms. The positive charge is completely delocalized on the

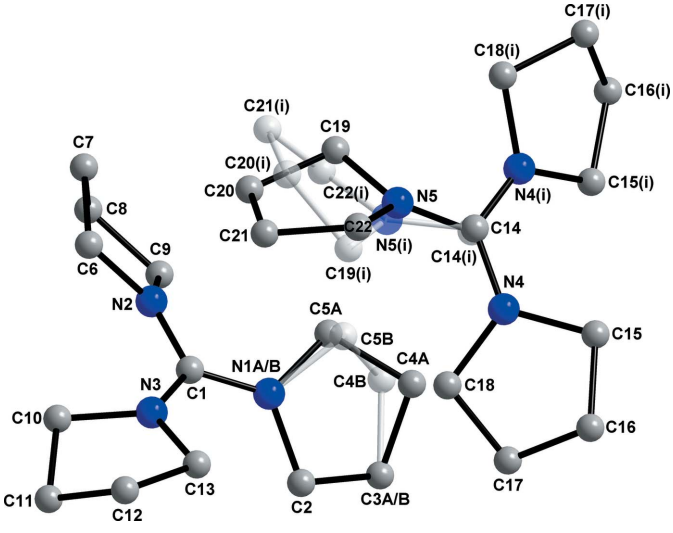


Figure 2
The tris(pyrrolidin-1-yl)carbenium ions in the crystal structure of the title compound. The methylene C atoms of the pyrrolidine ring (cation I) are disordered between the opaque and dark positions. The second disordered pyrrolidine moiety (cation II) exhibits two symmetry equivalent counterparts (dark and opaque positions). All H atoms have been omitted for clarity [symmetry code: (i) $-x, y, \frac{1}{2}-z$].

Table 1
Experimental details.

Crystal data	
Chemical formula	$(C_{13}H_{24}N_3)_3[Bi_2I_9]$
M_r	2227.11
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	100
a, *b*, *c* (Å)	14.1643 (7), 16.7047 (8), 24.9505 (12)
β (°)	91.601 (2)
V (Å^3)	5901.2 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	10.70
Crystal size (mm)	0.18 × 0.14 × 0.08
Data collection	
Diffractometer	Bruker Kappa APEXII DUO
Absorption correction	Multi-scan [Blessing, 1995; *SADABS* (Bruker, 2008)]
T_{min}, T_{max}	0.086, 0.276
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	61942, 9039, 8198
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.715
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.018, 0.034, 1.09
No. of reflections	9039
No. of parameters	314
No. of restraints	50
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.82, −0.96

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

CN_3 planes. The two Bi^{III} ions are coordinated by six iodide ions in a distorted octahedral manner, with the Bi—I bond lengths ranging from 2.9544 (2) to 3.2414 (2) Å. Two $[BiI_6]^{3-}$ octahedra are fused together through face-sharing, forming a dinuclear $[Bi_2I_9]^{3-}$ unit (Fig. 3). The bond lengths of bismuth to the terminal iodides [2.9544 (2)–2.9889 (2) Å] are shorter than the bridging ones [3.1450 (2)–3.2414 (2) Å]. The same anionic arrangement was observed in the crystal structure of $(CH_3NH_3)_3[Bi_2I_9]$, where the Bi—I bond lengths range from

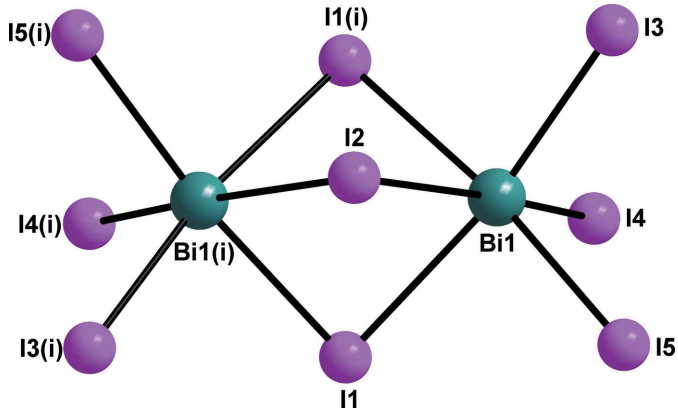


Figure 3
The $[Bi_2I_9]^{3-}$ ion in the crystal structure of the title compound [symmetry code: (i) $1-x, y, \frac{1}{2}-z$].

2.9529 (15) to 3.2286 (15) Å (Eckhardt *et al.*, 2016). Since no significant hydrogen bonding exists in the title compound, the crystal packing is dominated by electrostatic interactions between cations and anions.

Synthesis and crystallization

The title compound was obtained by mixing an aqueous solution of tris(pyrrolidin-1-yl)carbenium chloride with BiI_3/KI dissolved in water at room temperature. The orange colored precipitate was removed by filtration and washed with water and ethanol. The product was crystallized from an acetonitrile solution. After evaporation of the solvent at ambient temperature, orange single crystals suitable for X-ray analysis emerged.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The reflections 1 1 1 and 0 2 0 were affected by the beam stop and were omitted in the last steps of the refinement. The atoms C4 and C5 of cation I are disordered over two sets of sites (C4*A*/C4*B*, C5*A*/C5*B*) with refined occupancies of 0.757 (10):0.243 (10). The disordered pyrrolidine moiety at the cation II (N5, C14 and C19–C22) exhibits two half-occupied symmetry-equivalent counterparts related to each other by a twofold rotation axis. The two moieties of both disordered units were restrained to have similar geometries. The atoms N4, N5, C14, and $N4^{i}$ were restrained to be coplanar [symmetry operator: (i) $-x, y, \frac{1}{2} - z$]. The U^{ij} components of the ADPs of atoms of the second disordered pyrrolidine ring were restrained to be similar if closer than 1.7 Å, and carbon atom C14 was restrained to be close to isotropic.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160427 [doi:10.1107/S2414314616004272]

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Crystal data

$(C_{13}H_{24}N_3)_3[Bi_2I_9]$
M_r = 2227.11
Monoclinic, $C2/c$
a = 14.1643 (7) Å
b = 16.7047 (8) Å
c = 24.9505 (12) Å
β = 91.601 (2)°
V = 5901.2 (5) Å^3
Z = 4
$F(000)$ = 4048
D_x = 2.507 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 61942 reflections
θ = 1.6–30.5°
μ = 10.70 mm^{-1}
T = 100 K
Needle, orange
0.18 × 0.14 × 0.08 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} = 0.086, T_{max} = 0.276
61942 measured reflections
9039 independent reflections
8198 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 30.5°, θ_{min} = 1.6°
h = −17→20
k = −23→23
l = −35→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.034
S = 1.09
9039 reflections
314 parameters
50 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
$w = 1/[\sigma^2(F_o^2) + (0.0068P)^2 + 14.6985P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.000096 (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 $F^2 > \sigma(F^2)$ 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	0.53466 (2)	0.28574 (2)	0.16937 (2)	0.01601 (2)	
I1	0.35614 (2)	0.21736 (2)	0.22913 (2)	0.01759 (3)	
I2	0.5000	0.43436 (2)	0.2500	0.02276 (5)	
I3	0.71208 (2)	0.35570 (2)	0.13006 (2)	0.02981 (4)	
I4	0.55859 (2)	0.13174 (2)	0.11313 (2)	0.03154 (4)	
I5	0.41065 (2)	0.34792 (2)	0.07986 (2)	0.02754 (4)	
C1	0.03309 (17)	0.19805 (15)	0.06915 (9)	0.0210 (5)	
N2	−0.05065 (15)	0.16115 (13)	0.06500 (8)	0.0231 (4)	
C2	0.11229 (19)	0.32663 (15)	0.04243 (11)	0.0270 (5)	
H2A	0.1303	0.3037	0.0076	0.032*	
H2B	0.1689	0.3290	0.0665	0.032*	
N3	0.11203 (15)	0.15561 (12)	0.07760 (8)	0.0217 (4)	
C3A	0.0681 (2)	0.40961 (16)	0.03500 (12)	0.0320 (6)	0.757 (10)
H3A1	0.0353	0.4145	−0.0004	0.038*	0.757 (10)
H3A2	0.1164	0.4523	0.0386	0.038*	0.757 (10)
C4A	−0.0018 (3)	0.4134 (2)	0.08058 (19)	0.0313 (11)	0.757 (10)
H4A1	0.0310	0.4226	0.1156	0.038*	0.757 (10)
H4A2	−0.0496	0.4559	0.0743	0.038*	0.757 (10)
C5A	−0.0459 (3)	0.3310 (3)	0.0778 (3)	0.0286 (12)	0.757 (10)
H5A	−0.0746	0.3164	0.1122	0.034*	0.757 (10)
H5B	−0.0946	0.3276	0.0487	0.034*	0.757 (10)
N1A	0.03670 (15)	0.27905 (13)	0.06662 (9)	0.0256 (5)	0.757 (10)
C3B	0.0681 (2)	0.40961 (16)	0.03500 (12)	0.0320 (6)	0.243 (10)
H3B1	0.0697	0.4266	−0.0030	0.038*	0.243 (10)
H3B2	0.1020	0.4498	0.0575	0.038*	0.243 (10)
C4B	−0.0351 (8)	0.3993 (7)	0.0531 (6)	0.032 (4)	0.243 (10)
H4B1	−0.0600	0.4496	0.0682	0.039*	0.243 (10)
H4B2	−0.0771	0.3815	0.0230	0.039*	0.243 (10)
C5B	−0.0241 (11)	0.3358 (9)	0.0951 (5)	0.030 (4)	0.243 (10)
H5B1	0.0074	0.3565	0.1282	0.036*	0.243 (10)
H5B2	−0.0854	0.3115	0.1040	0.036*	0.243 (10)
N1B	0.03670 (15)	0.27905 (13)	0.06662 (9)	0.0256 (5)	0.243 (10)
C6	−0.0730 (2)	0.08320 (19)	0.08990 (14)	0.0390 (7)	
H6A	−0.0515	0.0816	0.1280	0.047*	
H6B	−0.0438	0.0383	0.0704	0.047*	
C7	−0.1801 (2)	0.0802 (2)	0.08463 (17)	0.0546 (10)	
H7A	−0.2037	0.0245	0.0858	0.065*	
H7B	−0.2099	0.1119	0.1131	0.065*	
C8	−0.1974 (2)	0.1170 (2)	0.03040 (15)	0.0481 (8)	
H8A	−0.2642	0.1335	0.0253	0.058*	

H8B	−0.1810	0.0793	0.0015	0.058*	
C9	−0.13268 (19)	0.18854 (18)	0.03134 (11)	0.0298 (6)	
H9A	−0.1131	0.2026	−0.0053	0.036*	
H9B	−0.1635	0.2355	0.0476	0.036*	
C10	0.12569 (18)	0.07202 (15)	0.05954 (10)	0.0222 (5)	
H10A	0.0965	0.0631	0.0235	0.027*	
H10B	0.0989	0.0334	0.0851	0.027*	
C11	0.23229 (18)	0.06521 (16)	0.05838 (11)	0.0268 (5)	
H11A	0.2573	0.0895	0.0255	0.032*	
H11B	0.2531	0.0087	0.0609	0.032*	
C12	0.2626 (2)	0.11214 (18)	0.10793 (13)	0.0354 (7)	
H12A	0.3304	0.1263	0.1073	0.042*	
H12B	0.2502	0.0818	0.1411	0.042*	
C13	0.2004 (2)	0.18620 (18)	0.10330 (13)	0.0360 (7)	
H13A	0.1884	0.2091	0.1391	0.043*	
H13B	0.2298	0.2276	0.0808	0.043*	
N4	0.07962 (15)	0.41124 (12)	0.23848 (10)	0.0267 (5)	
C15	0.10680 (18)	0.49321 (14)	0.25543 (12)	0.0266 (5)	
H15A	0.1011	0.5001	0.2946	0.032*	
H15B	0.0673	0.5339	0.2366	0.032*	
C16	0.20983 (19)	0.49899 (16)	0.23912 (12)	0.0303 (6)	
H16A	0.2270	0.5549	0.2305	0.036*	
H16B	0.2530	0.4789	0.2680	0.036*	
C17	0.2131 (2)	0.44610 (16)	0.18978 (12)	0.0315 (6)	
H17A	0.1842	0.4729	0.1580	0.038*	
H17B	0.2787	0.4305	0.1819	0.038*	
C18	0.1553 (2)	0.37471 (16)	0.20671 (13)	0.0340 (6)	
H18A	0.1286	0.3458	0.1751	0.041*	
H18B	0.1939	0.3371	0.2288	0.041*	
C14	0.0012 (8)	0.3727 (2)	0.25269 (17)	0.0282 (8)	0.5
N5	−0.0006 (11)	0.2916 (2)	0.2589 (3)	0.0230 (15)	0.5
C19	−0.0786 (8)	0.2412 (6)	0.2378 (3)	0.0259 (16)	0.5
H19A	−0.1137	0.2681	0.2080	0.031*	0.5
H19B	−0.1231	0.2272	0.2662	0.031*	0.5
C20	−0.0262 (4)	0.1674 (3)	0.2181 (2)	0.0303 (12)	0.5
H20A	−0.0631	0.1183	0.2251	0.036*	0.5
H20B	−0.0159	0.1712	0.1792	0.036*	0.5
C21	0.0675 (6)	0.1651 (4)	0.2490 (3)	0.0506 (17)	0.5
H21A	0.0708	0.1174	0.2725	0.061*	0.5
H21B	0.1204	0.1628	0.2240	0.061*	0.5
C22	0.0731 (8)	0.2391 (6)	0.2815 (3)	0.0278 (17)	0.5
H22A	0.0615	0.2273	0.3196	0.033*	0.5
H22B	0.1360	0.2643	0.2788	0.033*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Bi1	0.01522 (4)	0.01865 (4)	0.01416 (4)	0.00196 (3)	0.00050 (3)	0.00034 (3)

I1	0.01442 (7)	0.01826 (7)	0.01994 (7)	−0.00191 (5)	−0.00236 (5)	−0.00073 (5)
I2	0.02242 (11)	0.01260 (9)	0.03334 (12)	0.000	0.00212 (9)	0.000
I3	0.02103 (8)	0.03904 (10)	0.02977 (9)	−0.00113 (7)	0.00787 (7)	0.00943 (7)
I4	0.02613 (9)	0.03328 (9)	0.03497 (9)	0.00884 (7)	−0.00354 (7)	−0.01672 (7)
I5	0.02761 (9)	0.03426 (9)	0.02041 (8)	0.00227 (7)	−0.00543 (6)	0.00749 (6)
C1	0.0195 (12)	0.0265 (12)	0.0172 (11)	−0.0009 (9)	0.0021 (9)	0.0011 (9)
N2	0.0175 (10)	0.0281 (11)	0.0238 (10)	−0.0029 (8)	0.0004 (8)	0.0057 (8)
C2	0.0210 (13)	0.0258 (13)	0.0347 (14)	−0.0035 (10)	0.0086 (11)	−0.0045 (11)
N3	0.0178 (10)	0.0247 (10)	0.0223 (10)	−0.0013 (8)	−0.0036 (8)	−0.0014 (8)
C3A	0.0343 (16)	0.0239 (13)	0.0377 (15)	−0.0020 (11)	0.0030 (12)	−0.0039 (11)
C4A	0.025 (2)	0.031 (2)	0.037 (2)	0.0020 (15)	0.0003 (17)	−0.0142 (17)
C5A	0.019 (2)	0.026 (2)	0.040 (3)	−0.0004 (16)	0.003 (2)	−0.009 (2)
N1A	0.0201 (11)	0.0243 (11)	0.0327 (12)	−0.0011 (8)	0.0068 (9)	−0.0032 (9)
C3B	0.0343 (16)	0.0239 (13)	0.0377 (15)	−0.0020 (11)	0.0030 (12)	−0.0039 (11)
C4B	0.030 (7)	0.022 (6)	0.045 (9)	0.005 (5)	0.005 (6)	−0.007 (5)
C5B	0.018 (7)	0.048 (8)	0.025 (8)	0.002 (6)	−0.001 (5)	−0.015 (6)
N1B	0.0201 (11)	0.0243 (11)	0.0327 (12)	−0.0011 (8)	0.0068 (9)	−0.0032 (9)
C6	0.0251 (15)	0.0394 (17)	0.0528 (19)	−0.0031 (12)	0.0082 (13)	0.0194 (14)
C7	0.0236 (16)	0.055 (2)	0.086 (3)	−0.0070 (14)	0.0136 (17)	0.028 (2)
C8	0.0229 (16)	0.059 (2)	0.062 (2)	−0.0100 (14)	−0.0043 (15)	−0.0011 (18)
C9	0.0203 (13)	0.0414 (16)	0.0275 (13)	0.0003 (11)	−0.0038 (10)	0.0004 (12)
C10	0.0229 (12)	0.0225 (12)	0.0210 (11)	−0.0013 (9)	−0.0008 (9)	0.0025 (9)
C11	0.0224 (13)	0.0279 (13)	0.0301 (13)	0.0005 (10)	0.0013 (10)	−0.0010 (11)
C12	0.0245 (14)	0.0357 (15)	0.0450 (17)	0.0051 (12)	−0.0142 (12)	−0.0086 (13)
C13	0.0235 (14)	0.0335 (15)	0.0499 (18)	0.0024 (11)	−0.0166 (13)	−0.0121 (13)
N4	0.0208 (11)	0.0124 (9)	0.0471 (14)	0.0019 (8)	0.0030 (10)	−0.0024 (9)
C15	0.0204 (12)	0.0153 (11)	0.0441 (15)	−0.0035 (9)	0.0048 (11)	−0.0043 (10)
C16	0.0183 (12)	0.0235 (13)	0.0493 (17)	−0.0005 (10)	0.0045 (11)	−0.0019 (12)
C17	0.0215 (13)	0.0295 (14)	0.0437 (16)	0.0067 (11)	0.0052 (12)	−0.0025 (12)
C18	0.0258 (14)	0.0225 (13)	0.0539 (18)	0.0069 (10)	0.0047 (13)	−0.0082 (12)
C14	0.0231 (18)	0.0173 (16)	0.044 (2)	0.003 (4)	−0.0030 (17)	−0.003 (4)
N5	0.0240 (17)	0.0140 (14)	0.031 (4)	−0.004 (2)	−0.006 (4)	−0.0012 (17)
C19	0.030 (3)	0.019 (3)	0.029 (4)	−0.002 (2)	−0.004 (3)	−0.005 (3)
C20	0.037 (3)	0.020 (2)	0.033 (3)	0.004 (2)	−0.008 (2)	−0.016 (2)
C21	0.059 (4)	0.035 (3)	0.057 (4)	0.010 (3)	−0.019 (3)	−0.005 (3)
C22	0.027 (3)	0.017 (3)	0.038 (4)	0.005 (2)	−0.010 (4)	0.004 (4)

Geometric parameters (Å, º)

Bi1—I4	2.9544 (2)	C7—H7B	0.9900
Bi1—I3	2.9633 (2)	C8—C9	1.506 (4)
Bi1—I5	2.9889 (2)	C8—H8A	0.9900
Bi1—I1^{i}	3.1450 (2)	C8—H8B	0.9900
Bi1—I1	3.1832 (2)	C9—H9A	0.9900
Bi1—I2	3.2414 (2)	C9—H9B	0.9900
I1—Bi1^{i}	3.1450 (2)	C10—C11	1.515 (4)
I2—Bi1^{i}	3.2414 (2)	C10—H10A	0.9900
C1—N3	1.336 (3)	C10—H10B	0.9900

C1—N2	1.338 (3)	C11—C12	1.516 (4)
C1—N1B	1.356 (3)	C11—H11A	0.9900
C1—N1A	1.356 (3)	C11—H11B	0.9900
N2—C6	1.481 (3)	C12—C13	1.521 (4)
N2—C9	1.487 (3)	C12—H12A	0.9900
C2—N1B	1.476 (3)	C12—H12B	0.9900
C2—N1A	1.476 (3)	C13—H13A	0.9900
C2—C3A	1.530 (4)	C13—H13B	0.9900
C2—C3B	1.530 (4)	N4—C14	1.340 (10)
C2—H2A	0.9900	N4—C15	1.481 (3)
C2—H2B	0.9900	N4—C18	1.483 (3)
N3—C13	1.481 (3)	C15—C16	1.529 (4)
N3—C10	1.482 (3)	C15—H15A	0.9900
C3A—C4A	1.530 (5)	C15—H15B	0.9900
C3A—H3A1	0.9900	C16—C17	1.517 (4)
C3A—H3A2	0.9900	C16—H16A	0.9900
C4A—C5A	1.513 (6)	C16—H16B	0.9900
C4A—H4A1	0.9900	C17—C18	1.513 (4)
C4A—H4A2	0.9900	C17—H17A	0.9900
C5A—N1A	1.490 (5)	C17—H17B	0.9900
C5A—H5A	0.9900	C18—H18A	0.9900
C5A—H5B	0.9900	C18—H18B	0.9900
C3B—C4B	1.551 (11)	C14—N5	1.364 (5)
C3B—H3B1	0.9900	N5—C22	1.463 (14)
C3B—H3B2	0.9900	N5—C19	1.475 (14)
C4B—C5B	1.498 (14)	C19—C20	1.528 (11)
C4B—H4B1	0.9900	C19—H19A	0.9900
C4B—H4B2	0.9900	C19—H19B	0.9900
C5B—N1B	1.476 (13)	C20—C21	1.516 (8)
C5B—H5B1	0.9900	C20—H20A	0.9900
C5B—H5B2	0.9900	C20—H20B	0.9900
C6—C7	1.520 (4)	C21—C22	1.479 (12)
C6—H6A	0.9900	C21—H21A	0.9900
C6—H6B	0.9900	C21—H21B	0.9900
C7—C8	1.500 (5)	C22—H22A	0.9900
C7—H7A	0.9900	C22—H22B	0.9900

I4—Bi1—I3	94.391 (6)	C7—C8—H8A	111.1
I4—Bi1—I5	91.172 (6)	C9—C8—H8A	111.1
I3—Bi1—I5	95.836 (6)	C7—C8—H8B	111.1
I4—Bi1—I1^{i}	90.337 (6)	C9—C8—H8B	111.1
I3—Bi1—I1^{i}	90.414 (6)	H8A—C8—H8B	109.1
I5—Bi1—I1^{i}	173.438 (5)	N2—C9—C8	103.3 (2)
I4—Bi1—I1	90.759 (6)	N2—C9—H9A	111.1
I3—Bi1—I1	171.376 (5)	C8—C9—H9A	111.1
I5—Bi1—I1	90.962 (6)	N2—C9—H9B	111.1
I1^{i}—Bi1—I1	82.632 (6)	C8—C9—H9B	111.1
I4—Bi1—I2	169.447 (6)	H9A—C9—H9B	109.1

I3—Bi1—I2	92.746 (5)	N3—C10—C11	102.4 (2)
I5—Bi1—I2	95.829 (5)	N3—C10—H10A	111.3
I1^{i}—Bi1—I2	81.840 (5)	C11—C10—H10A	111.3
I1—Bi1—I2	81.260 (4)	N3—C10—H10B	111.3
Bi1^{i}—I1—Bi1	82.390 (5)	C11—C10—H10B	111.3
Bi1—I2—Bi1^{i}	80.022 (7)	H10A—C10—H10B	109.2
N3—C1—N2	120.3 (2)	C10—C11—C12	101.8 (2)
N3—C1—N1B	120.3 (2)	C10—C11—H11A	111.4
N2—C1—N1B	119.4 (2)	C12—C11—H11A	111.4
N3—C1—N1A	120.3 (2)	C10—C11—H11B	111.4
N2—C1—N1A	119.4 (2)	C12—C11—H11B	111.4
C1—N2—C6	124.9 (2)	H11A—C11—H11B	109.3
C1—N2—C9	125.4 (2)	C11—C12—C13	102.0 (2)
C6—N2—C9	109.6 (2)	C11—C12—H12A	111.4
N1A—C2—C3A	103.8 (2)	C13—C12—H12A	111.4
N1B—C2—C3B	103.8 (2)	C11—C12—H12B	111.4
N1A—C2—H2A	111.0	C13—C12—H12B	111.4
C3A—C2—H2A	111.0	H12A—C12—H12B	109.2
N1A—C2—H2B	111.0	N3—C13—C12	103.5 (2)
C3A—C2—H2B	111.0	N3—C13—H13A	111.1
H2A—C2—H2B	109.0	C12—C13—H13A	111.1
C1—N3—C13	125.4 (2)	N3—C13—H13B	111.1
C1—N3—C10	124.6 (2)	C12—C13—H13B	111.1
C13—N3—C10	109.9 (2)	H13A—C13—H13B	109.0
C2—C3A—C4A	102.6 (2)	C14—N4—C15	125.4 (3)
C2—C3A—H3A1	111.2	C14—N4—C18	124.1 (3)
C4A—C3A—H3A1	111.2	C15—N4—C18	110.3 (2)
C2—C3A—H3A2	111.2	N4—C15—C16	103.0 (2)
C4A—C3A—H3A2	111.2	N4—C15—H15A	111.2
H3A1—C3A—H3A2	109.2	C16—C15—H15A	111.2
C5A—C4A—C3A	101.8 (3)	N4—C15—H15B	111.2
C5A—C4A—H4A1	111.4	C16—C15—H15B	111.2
C3A—C4A—H4A1	111.4	H15A—C15—H15B	109.1
C5A—C4A—H4A2	111.4	C17—C16—C15	103.3 (2)
C3A—C4A—H4A2	111.4	C17—C16—H16A	111.1
H4A1—C4A—H4A2	109.3	C15—C16—H16A	111.1
N1A—C5A—C4A	102.3 (3)	C17—C16—H16B	111.1
N1A—C5A—H5A	111.3	C15—C16—H16B	111.1
C4A—C5A—H5A	111.3	H16A—C16—H16B	109.1
N1A—C5A—H5B	111.3	C18—C17—C16	101.7 (2)
C4A—C5A—H5B	111.3	C18—C17—H17A	111.4
H5A—C5A—H5B	109.2	C16—C17—H17A	111.4
C1—N1A—C2	125.8 (2)	C18—C17—H17B	111.4
C1—N1A—C5A	122.8 (3)	C16—C17—H17B	111.4
C2—N1A—C5A	110.2 (3)	H17A—C17—H17B	109.3
C2—C3B—C4B	104.5 (4)	N4—C18—C17	103.3 (2)
C2—C3B—H3B1	110.9	N4—C18—H18A	111.1
C4B—C3B—H3B1	110.9	C17—C18—H18A	111.1

C2—C3B—H3B2	110.9	N4—C18—H18B	111.1
C4B—C3B—H3B2	110.9	C17—C18—H18B	111.1
H3B1—C3B—H3B2	108.9	H18A—C18—H18B	109.1
C5B—C4B—C3B	101.6 (10)	N4—C14—N5	121.7 (9)
C5B—C4B—H4B1	111.4	C14—N5—C22	128.6 (10)
C3B—C4B—H4B1	111.4	C14—N5—C19	122.8 (9)
C5B—C4B—H4B2	111.4	C22—N5—C19	108.3 (3)
C3B—C4B—H4B2	111.4	N5—C19—C20	102.1 (8)
H4B1—C4B—H4B2	109.3	N5—C19—H19A	111.3
N1B—C5B—C4B	99.7 (9)	C20—C19—H19A	111.3
N1B—C5B—H5B1	111.8	N5—C19—H19B	111.3
C4B—C5B—H5B1	111.8	C20—C19—H19B	111.3
N1B—C5B—H5B2	111.8	H19A—C19—H19B	109.2
C4B—C5B—H5B2	111.8	C21—C20—C19	106.5 (6)
H5B1—C5B—H5B2	109.6	C21—C20—H20A	110.4
C1—N1B—C2	125.8 (2)	C19—C20—H20A	110.4
C1—N1B—C5B	126.5 (7)	C21—C20—H20B	110.4
C2—N1B—C5B	106.9 (7)	C19—C20—H20B	110.4
N2—C6—C7	102.6 (2)	H20A—C20—H20B	108.6
N2—C6—H6A	111.3	C22—C21—C20	106.9 (6)
C7—C6—H6A	111.3	C22—C21—H21A	110.3
N2—C6—H6B	111.3	C20—C21—H21A	110.3
C7—C6—H6B	111.3	C22—C21—H21B	110.3
H6A—C6—H6B	109.2	C20—C21—H21B	110.3
C8—C7—C6	101.7 (3)	H21A—C21—H21B	108.6
C8—C7—H7A	111.4	N5—C22—C21	105.3 (7)
C6—C7—H7A	111.4	N5—C22—H22A	110.7
C8—C7—H7B	111.4	C21—C22—H22A	110.7
C6—C7—H7B	111.4	N5—C22—H22B	110.7
H7A—C7—H7B	109.3	C21—C22—H22B	110.7
C7—C8—C9	103.1 (3)	H22A—C22—H22B	108.8

N3—C1—N2—C6	26.6 (4)	C4B—C5B—N1B—C2	46.0 (12)
N1B—C1—N2—C6	−151.0 (3)	C1—N2—C6—C7	166.3 (3)
N1A—C1—N2—C6	−151.0 (3)	C9—N2—C6—C7	−18.2 (3)
N3—C1—N2—C9	−148.2 (2)	N2—C6—C7—C8	38.2 (4)
N1B—C1—N2—C9	34.2 (4)	C6—C7—C8—C9	−44.6 (4)
N1A—C1—N2—C9	34.2 (4)	C1—N2—C9—C8	166.4 (3)
N2—C1—N3—C13	−154.8 (3)	C6—N2—C9—C8	−9.0 (3)
N1B—C1—N3—C13	22.9 (4)	C7—C8—C9—N2	33.2 (3)
N1A—C1—N3—C13	22.9 (4)	C1—N3—C10—C11	157.9 (2)
N2—C1—N3—C10	29.1 (3)	C13—N3—C10—C11	−18.7 (3)
N1B—C1—N3—C10	−153.3 (2)	N3—C10—C11—C12	39.0 (2)
N1A—C1—N3—C10	−153.3 (2)	C10—C11—C12—C13	−45.0 (3)
N1A—C2—C3A—C4A	29.4 (3)	C1—N3—C13—C12	174.4 (2)
C2—C3A—C4A—C5A	−43.2 (4)	C10—N3—C13—C12	−9.0 (3)
C3A—C4A—C5A—N1A	39.8 (5)	C11—C12—C13—N3	33.1 (3)
N3—C1—N1A—C2	37.1 (4)	C14—N4—C15—C16	167.3 (3)

N2—C1—N1A—C2	−145.3 (3)	C18—N4—C15—C16	−8.3 (3)
N3—C1—N1A—C5A	−156.4 (4)	N4—C15—C16—C17	31.5 (3)
N2—C1—N1A—C5A	21.3 (4)	C15—C16—C17—C18	−42.8 (3)
C3A—C2—N1A—C1	163.4 (2)	C14—N4—C18—C17	166.2 (3)
C3A—C2—N1A—C5A	−4.6 (4)	C15—N4—C18—C17	−18.2 (3)
C4A—C5A—N1A—C1	169.3 (3)	C16—C17—C18—N4	37.2 (3)
C4A—C5A—N1A—C2	−22.2 (5)	C15—N4—C14—N5	−147.8 (3)
N1B—C2—C3B—C4B	−3.7 (7)	C18—N4—C14—N5	27.2 (4)
C2—C3B—C4B—C5B	31.2 (12)	N4—C14—N5—C22	37.8 (9)
C3B—C4B—C5B—N1B	−46.1 (14)	N4—C14—N5—C19	−135.8 (8)
N3—C1—N1B—C2	37.1 (4)	C14—N5—C19—C20	141.3 (9)
N2—C1—N1B—C2	−145.3 (3)	C22—N5—C19—C20	−33.5 (8)
N3—C1—N1B—C5B	−131.7 (8)	N5—C19—C20—C21	23.0 (9)
N2—C1—N1B—C5B	46.0 (8)	C19—C20—C21—C22	−5.2 (10)
C3B—C2—N1B—C1	163.4 (2)	C14—N5—C22—C21	−143.3 (9)
C3B—C2—N1B—C5B	−26.1 (6)	C19—N5—C22—C21	31.1 (9)
C4B—C5B—N1B—C1	−143.5 (9)	C20—C21—C22—N5	−15.1 (10)

Symmetry code: (i) $-x+1, y, -z+1/2$.