

**Bis( $\mu$ -pyrrolidine-*N:N*)bis(dibromoboron), [(Br)<sub>2</sub>B(pyrrolidine)<sub>2</sub>B(Br)<sub>2</sub>]**Hijazi Abu Ali,<sup>a</sup> Israel Goldberg<sup>b\*</sup> and Morris Srebnik<sup>a</sup><sup>a</sup>Department of Medicinal Chemistry and Natural Products, School of Pharmacy, Hebrew University in Jerusalem, Jerusalem, Israel, and <sup>b</sup>School of Chemistry, Sackler Faculty of Exact Sciences, Tel-Aviv University, Ramat-Aviv, 69978 Tel-Aviv, Israel  
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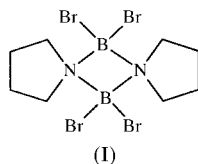
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The B atoms in the title compound, C<sub>8</sub>H<sub>16</sub>B<sub>2</sub>Br<sub>4</sub>N<sub>2</sub>, bridge between the two monomeric moieties, forming a (BN)<sub>2</sub> four-membered ring with partial bond orders of the B–N bonds.

**Comment**

The B<sub>2</sub>(NR<sub>2</sub>)<sub>2</sub> compounds are important intermediates in the synthesis of B<sub>2</sub>(OR)<sub>4</sub> compounds, which are very useful in the preparation of boronic acids from alkynes (Ishiyama, Matsuda *et al.*, 1996), 1,3-butadienes (Ishiyama, Yamamoto & Miyaura, 1996),  $\alpha,\beta$ -enones (Lawson *et al.*, 1997), methylenecyclopropanes (Ishiyama *et al.*, 1999) and aromatic compounds (Ishiyama *et al.*, 1995). The preparation of B<sub>2</sub>(NR<sub>2</sub>)<sub>2</sub> involves Wurtz coupling of the dialkylhaloboranes (Brotherton *et al.*, 1960; Nöth & Meister, 1961). Moreover, the structural aspects of diborane chemistry are not well explored and conformational problems are not yet properly understood (Moezzi *et al.*, 1992). It has been shown that the bromides (R<sub>2</sub>N)<sub>2</sub>BBR provide better yields than the chlorides (Brotherton *et al.*, 1960). In addition, the size of the R groups on the nitrogen influences the yields of B<sub>2</sub>(NR<sub>2</sub>)<sub>2</sub>. In an effort to synthesize B<sub>2</sub>(NR<sub>2</sub>)<sub>2</sub> compounds other than the methyl derivative and to explore their structural chemistry, we studied the dehalogenation of bis(pyrrolidino)bromoborane. During the course of this investigation, we isolated an unexpected by-product, *i.e.* the title compound, [(Br)<sub>2</sub>B(pyrrolidine)<sub>2</sub>B(Br)<sub>2</sub>], (I), in low yield. Subsequently, the same product was obtained in 90% yield, as described below.

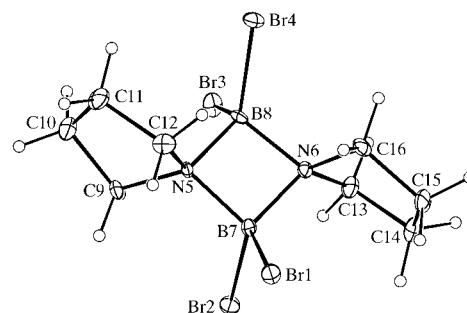


The molecular structure of (I) is illustrated in Fig. 1. The molecule has three possible directions for a C<sub>2</sub> axis, and it reveals an approximate C<sub>2</sub> symmetry about an axis passing perpendicular to the plane, and through the centre, of the four-membered ring. Its structural features, with relatively

long B–N bonds [mean 1.603 (7) Å], are similar to those observed in closely related aminoboron dichloride and difluoride dimers (Hazell, 1966; Edwards & Stadler, 1970; Jones, 1984; Clegg *et al.*, 1998; Jansen & Jäschke, 1999), as well as in other aminoboron dimers (Metzler & Nöth, 1995). This confirms that the observed elongation of the B–N bond is a genuine feature of the system.

The four-membered (BN)<sub>2</sub> ring in this structure has a distorted tetrahedral geometry. The N5–B7–N6 angle of 92.4 (2)° is significantly less than the ideal tetrahedral value. This results in a corresponding widening of the angles N5–B7–Br2 [113.5 (2)°], N6–B7–Br2 [113.7 (2)°], N5–B7–Br1 [113.9 (2)°] and N6–B7–Br1 [116.0 (2)°], while the Br2–B7–Br1 angle of 107.0 (2)° is relatively close to tetrahedral. The coordination geometry around the N atoms is also distorted from tetrahedral. Although the B7–N5–B8 angle of 86.8 (2)° is significantly less than the ideal tetrahedral value, widening of the C9–N5–C12 angle [100.3 (2)°] is not observed. On the other hand, the angles C9–N5–B7 [117.5 (2)°], C12–N5–B7 [118.0 (3)°], C9–N5–B8 [119.8 (3)°] and C12–N5–B8 [115.7 (2)°] are all expanded. Due to the presence of pseudo-C<sub>2</sub> symmetry, both B8 and N6 are similar to B7 and N5 in terms of geometry. The relatively long B–N bonds may be attributed to the coordination number of four around the B and N atoms, which, due to the bridging manner of the pyrrolidine groups, does not permit B–N  $\pi$ -bonding interactions, giving a bond order of about one. This is in contrast with an average B–N bond length of 1.48 Å observed in compounds having a B–N  $\pi$ -interaction and a bond order of about two (Nöth *et al.*, 1999). In addition, the electronegativity of the heterocyclic pyrrolidine groups also tends to inhibit  $\pi$ -bonding through their N atoms.

The four-membered heterocyclic ring has a bent nature. The dihedral angles between its N–B–N planes and between its B–N–B planes are 16.2 (3) and 15.4 (3)°, respectively. The pyrrolidine groups are puckered and adopt an envelope conformation with the N atoms at the flaps. The corresponding puckering parameters (Cremer & Pople, 1975) for the two rings are  $q_2 = 0.438$  (4) and 0.435 (3) Å, and  $\varphi_2 = -178.9$  (5) and  $-174.4$  (5)°. The carbon plane of one ring (C13–C16) points up, while the plane of the other ring (C9–C12) points down. The deviations of the N atoms from the respective

**Figure 1**

The molecular structure of (I), with displacement ellipsoids at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

carbon planes are 0.665 (3) Å for N5 and 0.655 (3) Å for N6. The H atoms of the pyrrolidine rings are eclipsed. The substituents of the four-membered ring are slightly staggered, with torsion angles of 12.7 (3)° for C16–N6–B8–Br4 and 16.0 (3)° for C13–N6–B8–Br3, to decrease eclipsing interactions between H and Br atoms.

## Experimental

A solution of boron tribromide (9.92 g, 0.04 mol) in pentane (30 ml) was added over a period of 20 min to a vigorously stirred solution of tris(pyrrolidino)borane (4.38 g, 0.20 mol) in pentane (100 ml) at 273 K. After an additional 3 h, the solution was allowed to warm to room temperature. The pentane was then removed by vacuum and the residue was distilled at 1 mm Hg (1 mm Hg = 133.322 Pa) to give the product as a clear liquid which, upon cooling below 273 K, gave colourless crystals of (I) in 90% yield (b.p. 403 K at 0.5 mm Hg). Analysis calculated for C<sub>8</sub>H<sub>16</sub>B<sub>4</sub>Br<sub>4</sub>N<sub>2</sub>: C 19.96, H 3.35, N 5.82, Br 66.38%; found: C 20.00, H 3.67, N 5.79, Br 66.26%. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded in CDCl<sub>3</sub> solution using a Varian Unity (300 or 75 MHz), with Me<sub>4</sub>Si as an internal standard. <sup>11</sup>B NMR spectra were recorded using a Bruker MSL-400 (128 MHz), with BF<sub>3</sub>·OEt<sub>2</sub> as an external standard. Spectroscopic analysis: <sup>1</sup>H NMR (CDCl<sub>3</sub>, δ, p.p.m.): 1.90 (t, 8H, CH<sub>2</sub>, <sup>3</sup>J<sub>H–H</sub> = 6.9 Hz), 3.45 (t, 8H, CH<sub>2</sub>N, <sup>3</sup>J<sub>H–H</sub> = 6.8 Hz); <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, δ, p.p.m.): 26.37 (CH<sub>2</sub>), 51.80 (CH<sub>2</sub>N); <sup>11</sup>B NMR (CDCl<sub>3</sub>, δ, p.p.m.): 23.23.

### Crystal data

C <sub>8</sub> H <sub>16</sub> B <sub>4</sub> Br <sub>4</sub> N <sub>2</sub>	$D_x = 2.316 \text{ Mg m}^{-3}$
$M_r = 481.49$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3271 reflections
$a = 12.8750 (2) \text{ \AA}$	$\theta = 2.6\text{--}27.9^\circ$
$b = 6.99600 (10) \text{ \AA}$	$\mu = 11.63 \text{ mm}^{-1}$
$c = 16.4480 (3) \text{ \AA}$	$T = 110 (2) \text{ K}$
$\beta = 111.2230 (8)^\circ$	Prism, colourless
$V = 1381.05 (4) \text{ \AA}^3$	$0.20 \times 0.15 \times 0.10 \text{ mm}$
$Z = 4$	

### Data collection

Nonius KappaCCD area-detector diffractometer	3271 independent reflections
$1^\circ \varphi$ scans	2850 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$R_{\text{int}} = 0.041$
$T_{\text{min}} = 0.155$ , $T_{\text{max}} = 0.341$	$\theta_{\text{max}} = 27.9^\circ$
5628 measured reflections	$h = 0 \rightarrow 16$
	$k = 0 \rightarrow 9$
	$l = -21 \rightarrow 20$

**Table 1**

Selected geometric parameters (Å, °).

Br1–B7	2.014 (4)	N5–B7	1.597 (4)
Br2–B7	1.990 (4)	N5–B8	1.610 (4)
Br3–B8	1.988 (4)	N6–B7	1.601 (4)
Br4–B8	2.006 (4)	N6–B8	1.604 (4)
B7–N5–B8	86.8 (2)	N5–B7–N6	92.4 (2)
B7–N6–B8	86.8 (2)	N6–B8–N5	91.8 (2)
B8–N5–B7–N6	11.2 (2)	B8–N6–B7–N5	–11.2 (2)
B8–N5–B7–Br2	–106.0 (2)	B8–N6–B7–Br2	105.8 (2)
B8–N5–B7–Br1	131.1 (2)	B8–N6–B7–Br1	–129.4 (2)

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 3.5877P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.069$	$(\Delta/\sigma)_{\text{max}} = 0.019$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$
3271 reflections	$\Delta\rho_{\text{min}} = -0.81 \text{ e \AA}^{-3}$
145 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997)
H-atom parameters constrained	Extinction coefficient: 0.00076 (17)

H atoms were refined as riding, with C–H = 0.99 Å and  $U_{\text{iso}} = 1.2U_{\text{eq}}$  of their parent atoms.

Data collection: COLLECT (Nonius, 1999); cell refinement: DENZO (Otwinowski, 1985); data reduction: DENZO; program(s) used to solve structure: DIRDIF96 (Beurskens *et al.*, 1996); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1117). Services for accessing these data are described at the back of the journal.

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## supporting information

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## Bis( $\mu$ -pyrrolidine-*N:N*)bis(dibromoboron), [(Br)<sub>2</sub>B(pyrrolidine)<sub>2</sub>B(Br)<sub>2</sub>]

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### Computing details

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski, 1985); data reduction: *DENZO*; program(s) used to solve structure: *DIRDIF96* (Beurskens *et al.*, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

### Bis( $\mu$ -pyrrolidine-*N:N*)bis(dibromoboron)

#### Crystal data

C<sub>8</sub>H<sub>16</sub>B<sub>2</sub>Br<sub>4</sub>N<sub>2</sub>

$M_r = 481.49$

Monoclinic, *P2<sub>1</sub>/c*

$a = 12.8750$  (2) Å

$b = 6.9960$  (1) Å

$c = 16.4480$  (3) Å

$\beta = 111.2230$  (8)°

$V = 1381.05$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 912$

$D_x = 2.316$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71070$  Å

Cell parameters from 3271 reflections

$\theta = 2.6$ – $27.9$ °

$\mu = 11.63$  mm<sup>-1</sup>

$T = 110$  K

Prism, colourless

$0.20 \times 0.15 \times 0.10$  mm

#### Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $56 \mu\text{m}$  pixels mm<sup>-1</sup>

1°  $\varphi$  scans

Absorption correction: multi-scan

(*SORTAV*; Blessing, 1995)

$T_{\text{min}} = 0.155$ ,  $T_{\text{max}} = 0.341$

5628 measured reflections

3271 independent reflections

2850 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\text{max}} = 27.9$ °,  $\theta_{\text{min}} = 2.6$ °

$h = 0 \rightarrow 16$

$k = 0 \rightarrow 9$

$l = -21 \rightarrow 20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.069$

$S = 1.01$

3271 reflections

145 parameters

0 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

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 3.5877P]$

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

$(\Delta/\sigma)_{\text{max}} = 0.019$

$\Delta\rho_{\text{max}} = 0.94$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.81$  e Å<sup>-3</sup>

Extinction correction: *SHELXL97* (Sheldrick, 1997),  $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00076 (17)

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.57268 (3)	0.10962 (5)	0.40957 (2)	0.01405 (9)
Br2	0.80023 (3)	0.03289 (5)	0.37569 (2)	0.01522 (10)
Br3	0.94374 (3)	-0.34596 (5)	0.56567 (2)	0.01505 (9)
Br4	0.75048 (3)	-0.37905 (5)	0.64711 (2)	0.01568 (9)
N5	0.7858 (2)	-0.0199 (4)	0.55275 (17)	0.0076 (5)
N6	0.7032 (2)	-0.2675 (4)	0.45883 (17)	0.0090 (5)
B7	0.7156 (3)	-0.0412 (5)	0.4509 (2)	0.0099 (7)
B8	0.7944 (3)	-0.2495 (5)	0.5556 (2)	0.0099 (7)
C9	0.8905 (3)	0.0988 (5)	0.5792 (2)	0.0126 (7)
H9A	0.8749	0.2272	0.5521	0.015*
H9B	0.9480	0.0359	0.5616	0.015*
C10	0.9284 (3)	0.1128 (5)	0.6782 (2)	0.0213 (8)
H10A	0.9654	0.2368	0.6991	0.026*
H10B	0.9810	0.0083	0.7065	0.026*
C11	0.8213 (3)	0.0950 (5)	0.6983 (2)	0.0192 (8)
H11A	0.8254	-0.0165	0.7365	0.023*
H11B	0.8088	0.2117	0.7276	0.023*
C12	0.7286 (3)	0.0689 (5)	0.6099 (2)	0.0147 (7)
H12A	0.6946	0.1933	0.5859	0.018*
H12B	0.6698	-0.0168	0.6145	0.018*
C13	0.7291 (3)	-0.3975 (5)	0.3953 (2)	0.0151 (7)
H13A	0.7484	-0.5275	0.4199	0.018*
H13B	0.7918	-0.3465	0.3806	0.018*
C14	0.6225 (3)	-0.4004 (5)	0.3155 (2)	0.0202 (8)
H14A	0.6141	-0.5239	0.2844	0.024*
H14B	0.6224	-0.2959	0.2749	0.024*
C15	0.5282 (3)	-0.3724 (5)	0.3511 (2)	0.0190 (8)
H15A	0.4815	-0.2609	0.3233	0.023*
H15B	0.4803	-0.4874	0.3404	0.023*
C16	0.5881 (3)	-0.3387 (5)	0.4495 (2)	0.0131 (7)
H16A	0.5482	-0.2421	0.4711	0.016*
H16B	0.5932	-0.4589	0.4825	0.016*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.01065 (17)	0.01145 (17)	0.01671 (17)	0.00229 (12)	0.00095 (13)	0.00177 (13)
Br2	0.01612 (18)	0.01887 (18)	0.01300 (16)	-0.00500 (13)	0.00805 (14)	0.00079 (13)
Br3	0.00910 (17)	0.01413 (17)	0.02053 (18)	0.00211 (12)	0.00371 (14)	-0.00210 (14)
Br4	0.0219 (2)	0.01401 (17)	0.01275 (17)	-0.00307 (13)	0.00820 (14)	0.00306 (13)
N5	0.0074 (13)	0.0055 (12)	0.0088 (12)	-0.0011 (10)	0.0015 (10)	-0.0011 (10)
N6	0.0087 (13)	0.0101 (13)	0.0089 (12)	-0.0020 (11)	0.0041 (11)	-0.0010 (11)
B7	0.0074 (17)	0.0110 (17)	0.0107 (16)	-0.0018 (14)	0.0026 (14)	-0.0004 (14)
B8	0.0127 (18)	0.0048 (16)	0.0119 (16)	0.0001 (14)	0.0041 (14)	0.0027 (14)
C9	0.0110 (16)	0.0105 (16)	0.0137 (16)	-0.0052 (13)	0.0015 (13)	-0.0003 (13)
C10	0.026 (2)	0.0181 (19)	0.0141 (17)	-0.0046 (16)	-0.0001 (15)	-0.0028 (15)
C11	0.029 (2)	0.0163 (17)	0.0108 (16)	0.0002 (15)	0.0049 (15)	-0.0036 (14)
C12	0.0194 (18)	0.0124 (16)	0.0154 (16)	0.0051 (14)	0.0101 (14)	-0.0005 (14)
C13	0.0177 (18)	0.0147 (17)	0.0118 (16)	-0.0019 (14)	0.0039 (14)	-0.0046 (14)
C14	0.024 (2)	0.0196 (19)	0.0135 (17)	-0.0071 (15)	0.0031 (15)	-0.0070 (15)
C15	0.0118 (18)	0.0179 (18)	0.0189 (18)	-0.0045 (14)	-0.0044 (14)	0.0007 (15)
C16	0.0095 (16)	0.0125 (16)	0.0169 (16)	-0.0028 (13)	0.0043 (13)	0.0033 (14)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Br1—B7	2.014 (4)	C10—H10B	0.9900
Br2—B7	1.990 (4)	C11—C12	1.523 (5)
Br3—B8	1.988 (4)	C11—H11A	0.9900
Br4—B8	2.006 (4)	C11—H11B	0.9900
N5—C9	1.507 (4)	C12—H12A	0.9900
N5—C12	1.521 (4)	C12—H12B	0.9900
N5—B7	1.597 (4)	C13—C14	1.517 (5)
N5—B8	1.610 (4)	C13—H13A	0.9900
N6—C13	1.511 (4)	C13—H13B	0.9900
N6—C16	1.517 (4)	C14—C15	1.540 (6)
N6—B7	1.601 (4)	C14—H14A	0.9900
N6—B8	1.604 (4)	C14—H14B	0.9900
C9—C10	1.525 (5)	C15—C16	1.537 (5)
C9—H9A	0.9900	C15—H15A	0.9900
C9—H9B	0.9900	C15—H15B	0.9900
C10—C11	1.535 (6)	C16—H16A	0.9900
C10—H10A	0.9900	C16—H16B	0.9900
C9—N5—C12	100.3 (2)	C12—C11—C10	105.0 (3)
C9—N5—B7	117.5 (2)	C12—C11—H11A	110.8
C12—N5—B7	118.0 (3)	C10—C11—H11A	110.8
C9—N5—B8	119.8 (3)	C12—C11—H11B	110.8
C12—N5—B8	115.7 (2)	C10—C11—H11B	110.8
B7—N5—B8	86.8 (2)	H11A—C11—H11B	108.8
C13—N6—C16	100.6 (2)	C11—C12—N5	104.6 (3)
C13—N6—B7	119.0 (2)	C11—C12—H12A	110.8

C16—N6—B7	116.1 (3)	N5—C12—H12A	110.8
C13—N6—B8	117.9 (3)	C11—C12—H12B	110.8
C16—N6—B8	117.6 (2)	N5—C12—H12B	110.8
B7—N6—B8	86.8 (2)	H12A—C12—H12B	108.9
N5—B7—N6	92.4 (2)	N6—C13—C14	104.4 (3)
N5—B7—Br2	113.5 (2)	N6—C13—H13A	110.9
N6—B7—Br2	113.7 (2)	C14—C13—H13A	110.9
N5—B7—Br1	113.9 (2)	N6—C13—H13B	110.9
N6—B7—Br1	116.0 (2)	C14—C13—H13B	110.9
Br2—B7—Br1	107.04 (17)	H13A—C13—H13B	108.9
N6—B8—N5	91.8 (2)	C13—C14—C15	105.1 (3)
N6—B8—Br3	113.3 (2)	C13—C14—H14A	110.7
N5—B8—Br3	113.3 (2)	C15—C14—H14A	110.7
N6—B8—Br4	113.9 (2)	C13—C14—H14B	110.7
N5—B8—Br4	115.9 (2)	C15—C14—H14B	110.7
Br3—B8—Br4	108.11 (17)	H14A—C14—H14B	108.8
N5—C9—C10	104.5 (3)	C16—C15—C14	104.8 (3)
N5—C9—H9A	110.9	C16—C15—H15A	110.8
C10—C9—H9A	110.9	C14—C15—H15A	110.8
N5—C9—H9B	110.9	C16—C15—H15B	110.8
C10—C9—H9B	110.9	C14—C15—H15B	110.8
H9A—C9—H9B	108.9	H15A—C15—H15B	108.9
C9—C10—C11	105.1 (3)	N6—C16—C15	104.9 (3)
C9—C10—H10A	110.7	N6—C16—H16A	110.8
C11—C10—H10A	110.7	C15—C16—H16A	110.8
C9—C10—H10B	110.7	N6—C16—H16B	110.8
C11—C10—H10B	110.7	C15—C16—H16B	110.8
H10A—C10—H10B	108.8	H16A—C16—H16B	108.8
C9—N5—B7—N6	133.3 (3)	C9—N5—B8—N6	-131.1 (3)
C12—N5—B7—N6	-106.3 (3)	C12—N5—B8—N6	108.6 (3)
B8—N5—B7—N6	11.2 (2)	B7—N5—B8—N6	-11.1 (2)
C9—N5—B7—Br2	16.1 (4)	C9—N5—B8—Br3	-14.8 (3)
C12—N5—B7—Br2	136.5 (2)	C12—N5—B8—Br3	-135.1 (2)
B8—N5—B7—Br2	-106.0 (2)	B7—N5—B8—Br3	105.2 (2)
C9—N5—B7—Br1	-106.8 (3)	C9—N5—B8—Br4	111.1 (3)
C12—N5—B7—Br1	13.6 (4)	C12—N5—B8—Br4	-9.2 (4)
B8—N5—B7—Br1	131.1 (2)	B7—N5—B8—Br4	-128.9 (2)
C13—N6—B7—N5	-131.5 (3)	C12—N5—C9—C10	43.4 (3)
C16—N6—B7—N5	108.1 (3)	B7—N5—C9—C10	172.7 (3)
B8—N6—B7—N5	-11.2 (2)	B8—N5—C9—C10	-84.3 (3)
C13—N6—B7—Br2	-14.5 (4)	N5—C9—C10—C11	-27.8 (3)
C16—N6—B7—Br2	-134.8 (2)	C9—C10—C11—C12	0.8 (4)
B8—N6—B7—Br2	105.8 (2)	C10—C11—C12—N5	26.2 (3)
C13—N6—B7—Br1	110.3 (3)	C9—N5—C12—C11	-42.9 (3)
C16—N6—B7—Br1	-10.0 (3)	B7—N5—C12—C11	-171.9 (3)
B8—N6—B7—Br1	-129.4 (2)	B8—N5—C12—C11	87.4 (3)
C13—N6—B8—N5	132.4 (3)	C16—N6—C13—C14	44.2 (3)

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C16—N6—B8—N5	-106.8 (3)	B7—N6—C13—C14	-83.8 (3)
B7—N6—B8—N5	11.1 (2)	B8—N6—C13—C14	173.5 (3)
C13—N6—B8—Br3	16.0 (3)	N6—C13—C14—C15	-30.4 (4)
C16—N6—B8—Br3	136.8 (2)	C13—C14—C15—C16	4.5 (4)
B7—N6—B8—Br3	-105.3 (2)	C13—N6—C16—C15	-41.2 (3)
C13—N6—B8—Br4	-108.1 (3)	B7—N6—C16—C15	88.7 (3)
C16—N6—B8—Br4	12.7 (3)	B8—N6—C16—C15	-170.6 (3)
B7—N6—B8—Br4	130.6 (2)	C14—C15—C16—N6	22.9 (3)

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