Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[*N*-(1-naphthyl)ethylenediammonium] hexabromidoplumbate(II)

Yao Chen, Ying-Ying Zheng, Gang Wu,* Mang Wang, Hong-Zheng Chen and Hui Yang

State Key Laboratory of Silicon Materials, Zhejiang University, Key Laboratory of Macromolecule Synthesis and Functionalization (Zhejiang University), Ministry of Education, Department of Material Science and Engineering, Zhejiang University, Hangzhou 310027, People's Republic of China
Correspondence e-mail: wmang@zju.edu.cn

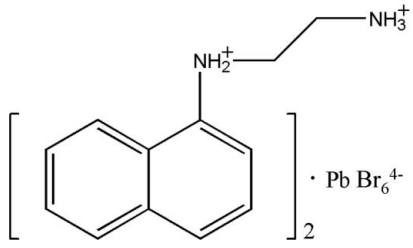
Received 8 January 2010; accepted 9 March 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.013 Å; R factor = 0.067; wR factor = 0.174; data-to-parameter ratio = 18.1.

The title compound, $(C_{12}H_{16}N_2)_2[PbBr_6]$, is an organic–inorganic salt, with two doubly protonated *N*-(1-naphthyl)-ethylenediammonium cations and one octahedral hexabromidoplumbate(II) anion. The Pb^{II} atom is located on a centre of inversion. The crystal structure consists of alternating inorganic and organic layers parallel to the *bc* plane. Face-to-face aromatic stacking interactions [centroid–centroid distance = 3.505 (5) Å] occur between parallel naphthalene systems in the organic layers, and N—H···Br hydrogen bonds between the cations and anions stabilize the crystal structure.

Related literature

For the related structure bis[*N*-(1-naphthyl)ethylenediammonium] hexaiodidoplumbate(II), see: Zheng *et al.* (2007).



Experimental

Crystal data

$(C_{12}H_{16}N_2)_2[PbBr_6]$
M_r = 1063.19
Triclinic, $P\overline{1}$
a = 8.1193 (4) Å
b = 8.5598 (4) Å
c = 12.4328 (6) Å
α = 80.4601 (13)°
β = 79.4756 (14)°
γ = 62.8592 (10)°
V = 752.63 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 13.59 mm^{-1}
T = 296 K
0.39 × 0.33 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.008, T_{max} = 0.066
6484 measured reflections
2938 independent reflections
2444 reflections with $I > 2\sigma(I)$
R_{int} = 0.092

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.174
S = 1.00
2938 reflections
162 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 3.73 e Å^{-3}
$\Delta\rho_{min}$ = −3.27 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pb1—Br1	3.0749 (8)	Pb1—Br3	3.0118 (10)
Pb1—Br2	2.9944 (10)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br1	0.90	2.48	3.364 (8)	168
N1—H1B···Br3^{i}	0.90	2.89	3.618 (7)	139
N2—H2B···Br2^{ii}	0.89	2.59	3.339 (9)	143

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

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the National Natural Science Foundation of China (Nos. 50990063, 50773067 and 50503021). The authors are grateful to Professor J.-M. Gu for assistance with the crystal structure analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2718).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2006). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2007). *CrystalStructure*. Rigaku Americas, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zheng, Y.-Y., Wu, G., Chen, H.-Z. & Wang, M. (2007). *Acta Cryst.* E**63**, m504–m506.

supporting information

Acta Cryst. (2010). E**66**, m417 [doi:10.1107/S1600536810008901]

Bis[*N*-(1-naphthyl)ethylenediammonium] hexabromidoplumbate(II)

Yao Chen, Ying-Ying Zheng, Gang Wu, Mang Wang, Hong-Zheng Chen and Hui Yang

S1. Comment

The title compound is a organic-inorganic compound, its structure is similar to bis(N-(1-naphthyl)ethylenediammonium) hexaiodoplumbate(II) (Zheng *et al.*, 2007). The crystal structure is composed of alternating organic and inorganic sheets nearly parallel to the *bc* plane (Fig. 1). The Pb^{II} cation is located on an inversion center and coordinated by six Br^- anions with a distorted octahedral geometry (Fig. 2). The Pb—Br bond lengths (Table 1) are in the range form 2.9944 (10) to 3.0749 (8) Å. The face-to-face distance between adjacent parallel naphthalene ring systems is 3.505 Å, indicating aromatic π-π interaction. The N—H···Br hydrogen bonding is present in the crystal structure (Table 2).

S2. Experimental

The N-(1-naphthyl)ethylenediamine hydrobromide and $PbBr_2$ are used as recieved. Concentrated hydrobromide and acetonitrile were degassed before using. All reactions were carried out under a nitrogen atmosphere. The title compound is prepared by a reaction of 0.1016 g N-(1-naphthyl)ethylenediamine hydrobromide with 0.0719 g $PbBr_2$ in the mixture solution of 14.6 ml hydrobromide and 1.8 ml acetonitrile at 353 K. The resulting solution was kept at 353 K for 1 h and then slowly cooled down to room temperature. The single crystals were filtered off from the solution.

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.93 (methylene), 0.97 (aromatic) and N—H = 0.89 or 0.90 Å, and included in the final cycles of the refinement in the riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(N)$.

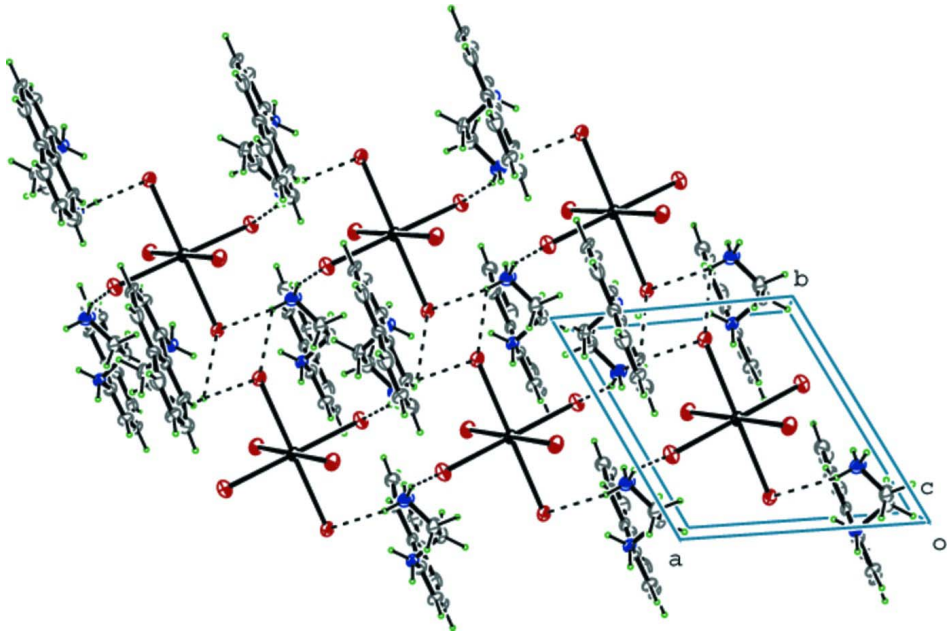


Figure 1

The molecular packing of the title compound viewed along the *b* axis.

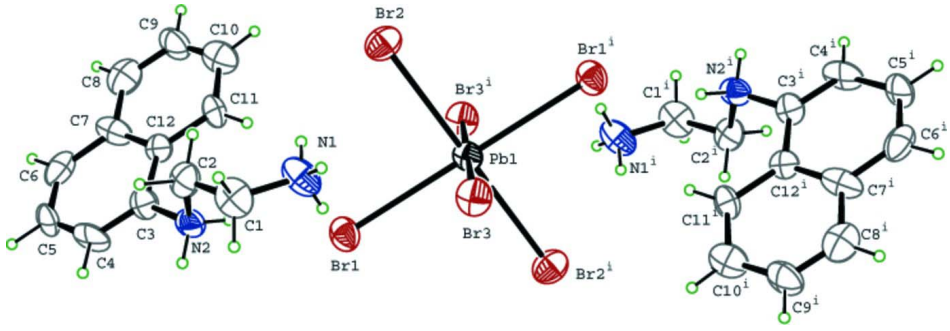


Figure 2

The structure of the title compound [symmetry code: (i) 1-x,1-y,1-z].

Bis[*N*-(1-naphthyl)ethylenediammonium] hexabromidoplumbate(II)

Crystal data

$(C_{12}H_{16}N_2)_2[PbBr_6]$
$M_r = 1063.19$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1193\ (4)$ Å
$b = 8.5598\ (4)$ Å
$c = 12.4328\ (6)$ Å
$\alpha = 80.4601\ (13)°$
$\beta = 79.4756\ (14)°$
$\gamma = 62.8592\ (10)°$
$V = 752.63\ (6)$ Å^3
$Z = 1$
$F(000) = 496$
$D_x = 2.346$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6259 reflections
$\theta = 3.0–27.4°$
$\mu = 13.59$ mm^{-1}
$T = 296$ K
Chunk, colourless
0.39 × 0.33 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: rolling anode
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.008, T_{max} = 0.066
6484 measured reflections
2938 independent reflections
2444 reflections with $I > 2\sigma(I)$
R_{int} = 0.092
θ_{max} = 26.0°, θ_{min} = 3.0°
h = −9→10
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.174
S = 1.00
2938 reflections
162 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
$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 3.73 e Å^{-3}
$\Delta\rho_{min}$ = −3.27 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0104 (12)

Special details

Experimental. Spectroscopic analysis: IR (KBr, cm^{-1}): 3008 (N—H asymmatric stretching), 2906 (N—H asymmatric stretching), 1573 (NH_2 bending), 1142 (CH_2 non-planar oscillating). Chemical analysis (calculated): C 27.09%, H 3.01%, N 5.27%; (found): C 27.12%, H 3.04%, N 5.23%.

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.5000	0.5000	0.5000	0.03697 (12)
Br3	0.69148 (13)	0.55171 (12)	0.66759 (8)	0.0503 (2)
Br2	0.85539 (12)	0.36179 (13)	0.34710 (8)	0.0533 (3)
Br1	0.44025 (12)	0.86889 (11)	0.39152 (8)	0.0482 (2)
N2	0.8772 (11)	0.7533 (10)	0.4518 (7)	0.054 (2)
H2A	0.7695	0.7973	0.4951	0.081*
H2B	0.9659	0.6741	0.4912	0.081*
H2C	0.8658	0.7018	0.3988	0.081*
N1	0.7162 (9)	1.0520 (8)	0.2540 (6)	0.0418 (17)
H1A	0.6464	0.9944	0.2816	0.050*
H1B	0.6673	1.1530	0.2863	0.050*

C1	0.7317 (10)	0.9604 (10)	0.0702 (7)	0.0371 (19)
C6	0.7273 (11)	1.0045 (10)	−0.0447 (8)	0.043 (2)
C10	0.7082 (12)	1.0940 (10)	0.1348 (8)	0.042 (2)
C2	0.7567 (12)	0.7896 (10)	0.1148 (8)	0.042 (2)
H2	0.7528	0.7596	0.1903	0.051*
C11	0.9145 (12)	0.9392 (12)	0.2815 (8)	0.047 (2)
H11A	0.9668	0.8306	0.2465	0.056*
H11B	0.9895	1.0014	0.2513	0.056*
C9	0.6906 (13)	1.2553 (10)	0.0898 (9)	0.050 (2)
H9	0.6822	1.3366	0.1343	0.059*
C5	0.7545 (13)	0.8761 (12)	−0.1131 (9)	0.051 (2)
H5	0.7519	0.9045	−0.1885	0.061*
C12	0.9269 (12)	0.8946 (12)	0.4031 (8)	0.051 (2)
H12A	1.0534	0.8616	0.4159	0.061*
H12B	0.8454	0.9995	0.4402	0.061*
C8	0.6851 (12)	1.2998 (10)	−0.0233 (8)	0.049 (2)
H8	0.6703	1.4115	−0.0540	0.058*
C4	0.7839 (13)	0.7129 (12)	−0.0684 (9)	0.056 (3)
H4	0.8025	0.6291	−0.1141	0.067*
C7	0.7014 (12)	1.1792 (12)	−0.0885 (8)	0.046 (2)
H7	0.6957	1.2109	−0.1635	0.055*
C3	0.7876 (13)	0.6654 (11)	0.0432 (9)	0.052 (3)
H3	0.8106	0.5506	0.0711	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.03482 (19)	0.03828 (18)	0.0450 (3)	−0.02084 (16)	−0.00683 (18)	−0.00605 (16)
Br3	0.0562 (4)	0.0602 (4)	0.0498 (5)	−0.0361 (4)	−0.0112 (4)	−0.0079 (4)
Br2	0.0390 (4)	0.0651 (5)	0.0514 (6)	−0.0190 (4)	−0.0035 (4)	−0.0078 (4)
Br1	0.0429 (4)	0.0463 (4)	0.0596 (6)	−0.0249 (3)	−0.0068 (4)	0.0014 (4)
N2	0.051 (4)	0.055 (4)	0.062 (5)	−0.028 (3)	−0.020 (4)	0.007 (4)
N1	0.040 (3)	0.039 (3)	0.054 (4)	−0.022 (3)	−0.003 (3)	−0.014 (3)
C1	0.029 (3)	0.042 (3)	0.046 (5)	−0.018 (3)	−0.001 (3)	−0.011 (3)
C6	0.029 (3)	0.040 (3)	0.063 (6)	−0.015 (3)	−0.012 (4)	−0.008 (4)
C10	0.041 (4)	0.045 (4)	0.045 (5)	−0.024 (3)	−0.006 (4)	−0.003 (3)
C2	0.049 (4)	0.042 (3)	0.048 (5)	−0.029 (3)	−0.009 (4)	−0.001 (3)
C11	0.039 (4)	0.052 (4)	0.057 (6)	−0.026 (4)	−0.001 (4)	−0.008 (4)
C9	0.055 (5)	0.037 (3)	0.066 (6)	−0.028 (3)	−0.006 (5)	−0.009 (4)
C5	0.046 (4)	0.063 (5)	0.053 (6)	−0.030 (4)	−0.009 (4)	−0.008 (4)
C12	0.051 (4)	0.054 (4)	0.063 (6)	−0.032 (4)	−0.022 (4)	0.000 (4)
C8	0.047 (4)	0.038 (4)	0.063 (6)	−0.025 (3)	−0.002 (4)	0.002 (4)
C4	0.053 (5)	0.055 (4)	0.070 (7)	−0.025 (4)	−0.008 (5)	−0.025 (4)
C7	0.038 (4)	0.058 (4)	0.038 (5)	−0.022 (4)	0.000 (4)	0.004 (4)
C3	0.045 (4)	0.045 (4)	0.071 (7)	−0.020 (4)	−0.009 (5)	−0.011 (4)

Geometric parameters (Å, º)

Pb1—Br1	3.0749 (8)	C10—C9	1.354 (12)
Pb1—Br1^{i}	3.0749 (8)	C2—C3	1.405 (13)
Pb1—Br2	2.9944 (10)	C2—H2	0.9300
Pb1—Br2^{i}	2.9944 (10)	C11—C12	1.506 (13)
Pb1—Br3^{i}	3.0118 (10)	C11—H11A	0.9700
Pb1—Br3	3.0118 (10)	C11—H11B	0.9700
N2—C12	1.451 (12)	C9—C8	1.399 (14)
N2—H2A	0.8900	C9—H9	0.9300
N2—H2B	0.8900	C5—C4	1.343 (14)
N2—H2C	0.8900	C5—H5	0.9300
N1—C10	1.472 (12)	C12—H12A	0.9700
N1—C11	1.522 (10)	C12—H12B	0.9700
N1—H1A	0.9000	C8—C7	1.361 (14)
N1—H1B	0.9000	C8—H8	0.9300
C1—C2	1.410 (11)	C4—C3	1.382 (15)
C1—C6	1.419 (13)	C4—H4	0.9300
C1—C10	1.428 (12)	C7—H7	0.9300
C6—C5	1.414 (13)	C3—H3	0.9300
C6—C7	1.436 (13)		
Br2—Pb1—Br2^{i}	180.0	C1—C10—N1	119.1 (7)
Br2—Pb1—Br3^{i}	88.45 (3)	C3—C2—C1	118.9 (9)
Br2^{i}—Pb1—Br3^{i}	91.55 (3)	C3—C2—H2	120.6
Br2—Pb1—Br3	91.55 (3)	C1—C2—H2	120.6
Br2^{i}—Pb1—Br3	88.45 (3)	C12—C11—N1	113.4 (7)
Br3^{i}—Pb1—Br3	180.0	C12—C11—H11A	108.9
Br2—Pb1—Br1	86.43 (3)	N1—C11—H11A	108.9
Br2^{i}—Pb1—Br1	93.57 (3)	C12—C11—H11B	108.9
Br3^{i}—Pb1—Br1	92.47 (3)	N1—C11—H11B	108.9
Br3—Pb1—Br1	87.53 (3)	H11A—C11—H11B	107.7
Br2—Pb1—Br1^{i}	93.57 (3)	C10—C9—C8	120.1 (9)
Br2^{i}—Pb1—Br1^{i}	86.43 (3)	C10—C9—H9	119.9
Br3^{i}—Pb1—Br1^{i}	87.53 (3)	C8—C9—H9	119.9
Br3—Pb1—Br1^{i}	92.47 (3)	C4—C5—C6	119.6 (10)
Br1—Pb1—Br1^{i}	180.0	C4—C5—H5	120.2
C12—N2—H2A	109.5	C6—C5—H5	120.2
C12—N2—H2B	109.5	N2—C12—C11	114.6 (8)
H2A—N2—H2B	109.5	N2—C12—H12A	108.6
C12—N2—H2C	109.5	C11—C12—H12A	108.6
H2A—N2—H2C	109.5	N2—C12—H12B	108.6
H2B—N2—H2C	109.5	C11—C12—H12B	108.6
C10—N1—C11	112.3 (7)	H12A—C12—H12B	107.6
C10—N1—H1A	109.2	C7—C8—C9	119.8 (8)
C11—N1—H1A	109.2	C7—C8—H8	120.1
C10—N1—H1B	109.2	C9—C8—H8	120.1
C11—N1—H1B	109.2	C5—C4—C3	122.1 (10)

H1A—N1—H1B	107.9	C5—C4—H4	118.9
C2—C1—C6	119.0 (8)	C3—C4—H4	118.9
C2—C1—C10	123.5 (8)	C8—C7—C6	121.8 (9)
C6—C1—C10	117.5 (7)	C8—C7—H7	119.1
C5—C6—C1	120.0 (8)	C6—C7—H7	119.1
C5—C6—C7	121.9 (9)	C4—C3—C2	120.4 (9)
C1—C6—C7	118.1 (8)	C4—C3—H3	119.8
C9—C10—C1	122.5 (9)	C2—C3—H3	119.8
C9—C10—N1	118.2 (8)		

C2—C1—C6—C5	−2.2 (12)	C1—C10—C9—C8	3.4 (14)
C10—C1—C6—C5	178.4 (8)	N1—C10—C9—C8	179.1 (8)
C2—C1—C6—C7	179.9 (8)	C1—C6—C5—C4	0.1 (13)
C10—C1—C6—C7	0.5 (11)	C7—C6—C5—C4	177.9 (9)
C2—C1—C10—C9	177.7 (8)	N1—C11—C12—N2	78.7 (10)
C6—C1—C10—C9	−2.9 (12)	C10—C9—C8—C7	−1.4 (14)
C2—C1—C10—N1	2.1 (12)	C6—C5—C4—C3	0.4 (15)
C6—C1—C10—N1	−178.6 (7)	C9—C8—C7—C6	−1.0 (13)
C11—N1—C10—C9	−100.8 (9)	C5—C6—C7—C8	−176.5 (8)
C11—N1—C10—C1	75.0 (10)	C1—C6—C7—C8	1.3 (12)
C6—C1—C2—C3	3.7 (12)	C5—C4—C3—C2	1.2 (15)
C10—C1—C2—C3	−177.0 (8)	C1—C2—C3—C4	−3.2 (13)
C10—N1—C11—C12	179.9 (7)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1	0.90	2.48	3.364 (8)	168
N1—H1*B*···Br3^{ii}	0.90	2.89	3.618 (7)	139
N2—H2*B*···Br2^{iii}	0.89	2.59	3.339 (9)	143

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