Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# Hydrogen bonding in (2-bromobenzyl)-dimethylammonium bromide

**Richard A. Varga* and Cristian Silvestru**

Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, Arany Janos Str. no. 11, RO-400028, Cluj Napoca, Romania
Correspondence e-mail: richy@chem.ubbcluj.ro

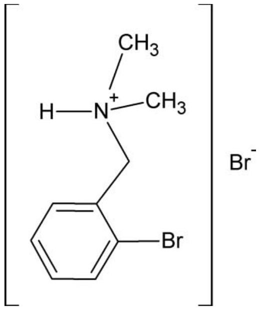
Received 22 June 2007; accepted 29 June 2007

Key indicators: single-crystal X-ray study; $T$ = 297 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.049; $wR$ factor = 0.110; data-to-parameter ratio = 17.0.

Ions of the title compound, $C_9H_{13}BrN^+\cdot Br^-$, are linked by a strong N—H···Br$^-$ hydrogen bond, and this unit is further linked to another three anion–cation units by weak C—H···Br$^-$ contacts (H···Br range 2.95–3.09 Å), generating a two-dimensional layer parallel to the $bc$ plane. The layers are stacked along the $a$ axis without any interactions in the third dimension. The Br atom of the cation forms only an intramolecular hydrogen bond (H···Br = 2.76 Å) with one of the methylene H atoms.

## Related literature

For similar supramolecular structures formed through H···Br contacts, see: Varga *et al.* (2006); Varga & Silvestru (2007).



## Experimental

### Crystal data

$C_9H_{13}BrN^+\cdot Br^-$
$M_r$ = 295.02
Monoclinic, $P2_1/c$
$a$ = 8.6227 (8) Å
$b$ = 14.6503 (13) Å
$c$ = 9.2118 (8) Å
$\beta$ = 107.339 (2)°
$V$ = 1110.80 (17) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 7.25 mm$^{-1}$
$T$ = 297 (2) K
0.23 × 0.21 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SHELXTL*; Bruker, 2001)
$T_{min}$ = 0.216, $T_{max}$ = 0.491
7866 measured reflections
1957 independent reflections
1700 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.110
$S$ = 1.17
1957 reflections
115 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.70 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.51 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···Br2 | 0.86 (2) | 2.38 (2) | 3.217 (4) | 165 (6) |
| C7—H7$B$···Br1 | 0.97 | 2.76 | 3.266 (5) | 113 |
| C8—H8$A$···Br2$^{i}$ | 0.96 | 3.04 | 3.916 (6) | 153 |
| C7—H7$B$···Br2$^{i}$ | 0.97 | 3.09 | 3.918 (6) | 144 |
| C7—H7$A$···Br2$^{ii}$ | 0.97 | 2.95 | 3.819 (5) | 150 |
| C8—H8$B$···Br2$^{ii}$ | 0.96 | 3.02 | 3.895 (7) | 152 |

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, -y+2, -z+2$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2007).

We thank the National Center for X-ray Diffraction ('Babes-Bolyai' University, Cluj-Napoca) for performing the single-crystal X-ray diffraction study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2024).

## References

Brandenburg, K. & Putz, H. (2006). *DIAMOND*. Version 3. Crystal Impact GbR, Bonn, Germany.
Bruker (2000). *SMART* (Version 5.625) and *SAINT-Plus* (Version 6.29). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SHELXTL*. Version 6.10.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Varga, R. A., Rotar, A., Schuermann, M., Jurkschat, K. & Silvestru, C. (2006). *Eur. J. Inorg. Chem.* **7**, 1475–1486.
Varga, R. A. & Silvestru, C. (2007). *Acta Cryst.* **C63**, m48–m50.
Westrip, S. P. (2007). *publCIF*. In preparation.

doi:10.1107/S1600536807031947

# supplementary materials

*Acta Cryst.* (2007). E**63**, o3381    [ **doi:10.1107/S1600536807031947** ]

# Hydrogen bonding in (2-bromobenzyl)dimethylammonium bromide

**R. A. Varga** and **C. Silvestru**

## Comment

In solid state, the anion-cation unit of the title compound is held together by a strong N—H···Br$^-$ hydrogen bond [H1···Br2 = 2.83 Å] (Fig. 1). Another intramolecular H···Br contact is present between one hydrogen from the methylene group and the bromine bonded to the aromatic ring [H7B···Br1 = 2.76 Å]. The resulting C7—H7B···Br1 angle has a small value due to the rigid skeleton bearing both the donor and the acceptor part of the hydrogen bond-type contact.

The anion-cation unit forms eight weak intermolecular contacts through the two methylene hydrogen atoms, two other H atoms, each from one methyl group bonded to nitrogen, and the bromine anion with three neighboring units (Fig. 2). Four of these interactions are with another anion-cation unit result in zigzag polymers along the *c* axis and the remaining four contacts with two other units link the one-dimensional arrays, along the *b* axis, in a two-dimensional supramolecular layer-type arrangement (Varga *et al.*, 2006; Varga & Silvestru, 2007) (Fig. 3). The layers are stacked together, with the aromatic rings intercalated, but with no contacts of any type on the third dimension (Fig. 4).

## Experimental

The title compound was obtained unintentionally as the decomposition product from the reaction between dibromo[2-(dimethylaminomethyl)phenyl][2-(dimethylammoniomethyl)phenyl]tin(IV) tetrabromo[2-(dimethylaminomethyl)phenyl]tin(IV) and excess of potassium hydroxide in a $CHCl_3$ / $H_2O$ (1:1) mixture. Crystals suitable for single-crystal X-ray diffraction were obtained from a $CHCl_3$-*n*-hexane (1:4) mixture.

## Refinement

All hydrogen atoms were placed in calculated positions using a riding model, with C—H = 0.93–0.97 Å and with $U_{iso}$= 1.5$U_{eq}$ (C) for methyl H and $U_{iso}$= 1.2$U_{eq}$ (C) for aryl H. The methyl groups were allowed to rotate but not to tip. The hydrogen H1 atom bonded to N1 was found in a difference map and refined with a restrained N—H distance of 0.86 (2) Å

## Figures

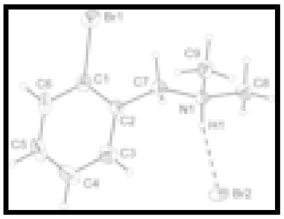


Fig. 1. View of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bond shown with dashed line.

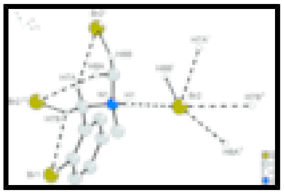


Fig. 2. The hydrogen bonding (dashed lines) in structure of the title compound. [Symmetry codes: (i) $2 - x$, $2 - y$, $2 - z$; (ii) $2 - x$, $1/2 + y$, $1.5 - z$; (iii) $2 - x$, $-1/2 + y$, $1.5 - z$.]

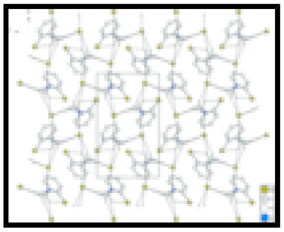


Fig. 3. Supramolecular arrangement of the title compound (hydrogen bonds are shown as dashed lines), view along *a* axis.

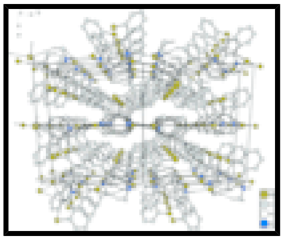


Fig. 4. Crystal packing of the title compound along the *c* axis.

### (2-bromobenzyl)dimethylammonium bromide

*Crystal data*

$C_9H_{13}BrN^+\cdot Br^-$

$M_r = 295.02$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.6227$ (8) Å

$b = 14.6503$ (13) Å

$c = 9.2118$ (8) Å

$\beta = 107.339$ (2)°

$V = 1110.80$ (17) Å$^3$

$Z = 4$

$F_{000} = 576$

$D_x = 1.764$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3587 reflections

$\theta = 2.5$–27.6°

$\mu = 7.25$ mm$^{-1}$

$T = 297$ (2) K

Block, colourless

0.23 × 0.21 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 297(2)$ K

$\varphi$ and $\omega$ scans

Absorption correction: multi-scan (SHELXTL; Bruker, 2001)

$T_{\min} = 0.216$, $T_{\max} = 0.491$

1957 independent reflections

1700 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 25.0$°

$\theta_{\min} = 2.5$°

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 17$

7866 measured reflections

$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.110$

$S = 1.17$

1957 reflections

115 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2 + 2.3199P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.70$ e Å$^{-3}$

$\Delta\rho_{min} = -0.51$ e Å$^{-3}$

Extinction correction: none

*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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br2 | 1.01040 (8) | 1.09349 (4) | 0.77395 (7) | 0.0421 (2) |
| Br1 | 0.55425 (9) | 0.75580 (5) | 0.50628 (9) | 0.0672 (3) |
| C1 | 0.5417 (7) | 0.8600 (4) | 0.6257 (7) | 0.0390 (13) |
| C2 | 0.6691 (6) | 0.8827 (3) | 0.7517 (6) | 0.0301 (12) |
| C3 | 0.6466 (8) | 0.9588 (4) | 0.8348 (7) | 0.0458 (15) |
| H3 | 0.7295 | 0.9760 | 0.9209 | 0.055* |
| C4 | 0.5061 (9) | 1.0087 (5) | 0.7931 (9) | 0.0605 (19) |
| H4 | 0.4941 | 1.0589 | 0.8505 | 0.073* |
| C5 | 0.3849 (8) | 0.9846 (5) | 0.6678 (9) | 0.0558 (18) |
| H5 | 0.2898 | 1.0188 | 0.6394 | 0.067* |
| C6 | 0.4001 (7) | 0.9102 (5) | 0.5814 (8) | 0.0509 (16) |
| H6 | 0.3166 | 0.8941 | 0.4950 | 0.061* |
| C7 | 0.8256 (6) | 0.8306 (4) | 0.8117 (6) | 0.0353 (13) |
| H7A | 0.8518 | 0.8256 | 0.9214 | 0.042* |
| H7B | 0.8110 | 0.7694 | 0.7697 | 0.042* |

| | | | | |
|---|---|---|---|---|
| C8 | 1.1244 (7) | 0.8408 (4) | 0.8720 (7) | 0.0432 (14) |
| H8A | 1.1317 | 0.7763 | 0.8568 | 0.065* |
| H8B | 1.1327 | 0.8525 | 0.9766 | 0.065* |
| H8C | 1.2111 | 0.8714 | 0.8463 | 0.065* |
| C9 | 0.9548 (7) | 0.8636 (4) | 0.6101 (6) | 0.0437 (14) |
| H9A | 1.0388 | 0.8987 | 0.5876 | 0.066* |
| H9B | 0.8506 | 0.8841 | 0.5474 | 0.066* |
| H9C | 0.9683 | 0.8002 | 0.5901 | 0.066* |
| N1 | 0.9650 (5) | 0.8755 (3) | 0.7728 (5) | 0.0311 (10) |
| H1 | 0.964 (8) | 0.9336 (15) | 0.783 (8) | 0.06 (2)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br2 | 0.0564 (4) | 0.0284 (3) | 0.0410 (3) | −0.0039 (3) | 0.0136 (3) | −0.0004 (2) |
| Br1 | 0.0509 (5) | 0.0714 (5) | 0.0772 (6) | −0.0147 (4) | 0.0156 (4) | −0.0333 (4) |
| C1 | 0.036 (3) | 0.041 (3) | 0.042 (3) | −0.004 (3) | 0.014 (3) | 0.003 (3) |
| C2 | 0.031 (3) | 0.027 (3) | 0.034 (3) | 0.001 (2) | 0.013 (2) | 0.004 (2) |
| C3 | 0.047 (4) | 0.052 (4) | 0.040 (3) | 0.001 (3) | 0.016 (3) | 0.001 (3) |
| C4 | 0.072 (5) | 0.046 (4) | 0.075 (5) | 0.017 (4) | 0.041 (4) | 0.004 (3) |
| C5 | 0.039 (4) | 0.060 (4) | 0.076 (5) | 0.020 (3) | 0.029 (4) | 0.027 (4) |
| C6 | 0.025 (3) | 0.072 (5) | 0.055 (4) | 0.002 (3) | 0.010 (3) | 0.015 (3) |
| C7 | 0.035 (3) | 0.033 (3) | 0.039 (3) | 0.001 (2) | 0.011 (3) | 0.004 (2) |
| C8 | 0.035 (3) | 0.042 (3) | 0.049 (4) | 0.007 (3) | 0.008 (3) | 0.003 (3) |
| C9 | 0.041 (3) | 0.055 (4) | 0.037 (3) | −0.008 (3) | 0.014 (3) | −0.004 (3) |
| N1 | 0.030 (2) | 0.026 (2) | 0.038 (3) | −0.0002 (19) | 0.011 (2) | 0.0012 (19) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Br2—H1 | 2.38 (3) | C7—N1 | 1.503 (7) |
| Br1—C1 | 1.904 (6) | C7—H7A | 0.9700 |
| C1—C6 | 1.378 (8) | C7—H7B | 0.9700 |
| C1—C2 | 1.380 (8) | C8—N1 | 1.494 (7) |
| C2—C3 | 1.398 (8) | C8—H8A | 0.9600 |
| C2—C7 | 1.504 (7) | C8—H8B | 0.9600 |
| C3—C4 | 1.368 (9) | C8—H8C | 0.9600 |
| C3—H3 | 0.9300 | C9—N1 | 1.485 (7) |
| C4—C5 | 1.353 (10) | C9—H9A | 0.9600 |
| C4—H4 | 0.9300 | C9—H9B | 0.9600 |
| C5—C6 | 1.379 (10) | C9—H9C | 0.9600 |
| C5—H5 | 0.9300 | N1—H1 | 0.86 (2) |
| C6—H6 | 0.9300 | | |
| C6—C1—C2 | 122.3 (6) | N1—C7—H7B | 109.1 |
| C6—C1—Br1 | 117.0 (5) | C2—C7—H7B | 109.1 |
| C2—C1—Br1 | 120.7 (4) | H7A—C7—H7B | 107.8 |
| C1—C2—C3 | 116.5 (5) | N1—C8—H8A | 109.5 |
| C1—C2—C7 | 126.1 (5) | N1—C8—H8B | 109.5 |
| C3—C2—C7 | 117.3 (5) | H8A—C8—H8B | 109.5 |

| | | | |
|---|---|---|---|
| C4—C3—C2 | 121.9 (6) | N1—C8—H8C | 109.5 |
| C4—C3—H3 | 119.1 | H8A—C8—H8C | 109.5 |
| C2—C3—H3 | 119.1 | H8B—C8—H8C | 109.5 |
| C5—C4—C3 | 119.6 (7) | N1—C9—H9A | 109.5 |
| C5—C4—H4 | 120.2 | N1—C9—H9B | 109.5 |
| C3—C4—H4 | 120.2 | H9A—C9—H9B | 109.5 |
| C4—C5—C6 | 121.1 (6) | N1—C9—H9C | 109.5 |
| C4—C5—H5 | 119.4 | H9A—C9—H9C | 109.5 |
| C6—C5—H5 | 119.4 | H9B—C9—H9C | 109.5 |
| C1—C6—C5 | 118.6 (6) | C9—N1—C8 | 110.1 (4) |
| C1—C6—H6 | 120.7 | C9—N1—C7 | 112.2 (4) |
| C5—C6—H6 | 120.7 | C8—N1—C7 | 111.2 (4) |
| N1—C7—C2 | 112.6 (4) | C9—N1—H1 | 103 (5) |
| N1—C7—H7A | 109.1 | C8—N1—H1 | 108 (5) |
| C2—C7—H7A | 109.1 | C7—N1—H1 | 112 (5) |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 1.0 (8) | C2—C1—C6—C5 | −1.0 (9) |
| Br1—C1—C2—C3 | −178.2 (4) | Br1—C1—C6—C5 | 178.2 (5) |
| C6—C1—C2—C7 | 178.0 (5) | C4—C5—C6—C1 | 0.4 (10) |
| Br1—C1—C2—C7 | −1.2 (8) | C1—C2—C7—N1 | 103.2 (6) |
| C1—C2—C3—C4 | −0.4 (8) | C3—C2—C7—N1 | −79.9 (6) |
| C7—C2—C3—C4 | −177.6 (5) | C2—C7—N1—C9 | −74.2 (6) |
| C2—C3—C4—C5 | −0.2 (10) | C2—C7—N1—C8 | 162.0 (5) |
| C3—C4—C5—C6 | 0.2 (10) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Br2 | 0.86 (2) | 2.38 (2) | 3.217 (4) | 165 (6) |
| C7—H7B···Br1 | 0.97 | 2.76 | 3.266 (5) | 113 |
| C8—H8A···Br2$^{i}$ | 0.96 | 3.04 | 3.916 (6) | 153 |
| C7—H7B···Br2$^{i}$ | 0.97 | 3.09 | 3.918 (6) | 144 |
| C7—H7A···Br2$^{ii}$ | 0.97 | 2.95 | 3.819 (5) | 150 |
| C8—H8B···Br2$^{ii}$ | 0.96 | 3.02 | 3.895 (7) | 152 |

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) −*x*+2, −*y*+2, −*z*+2.

Fig. 1

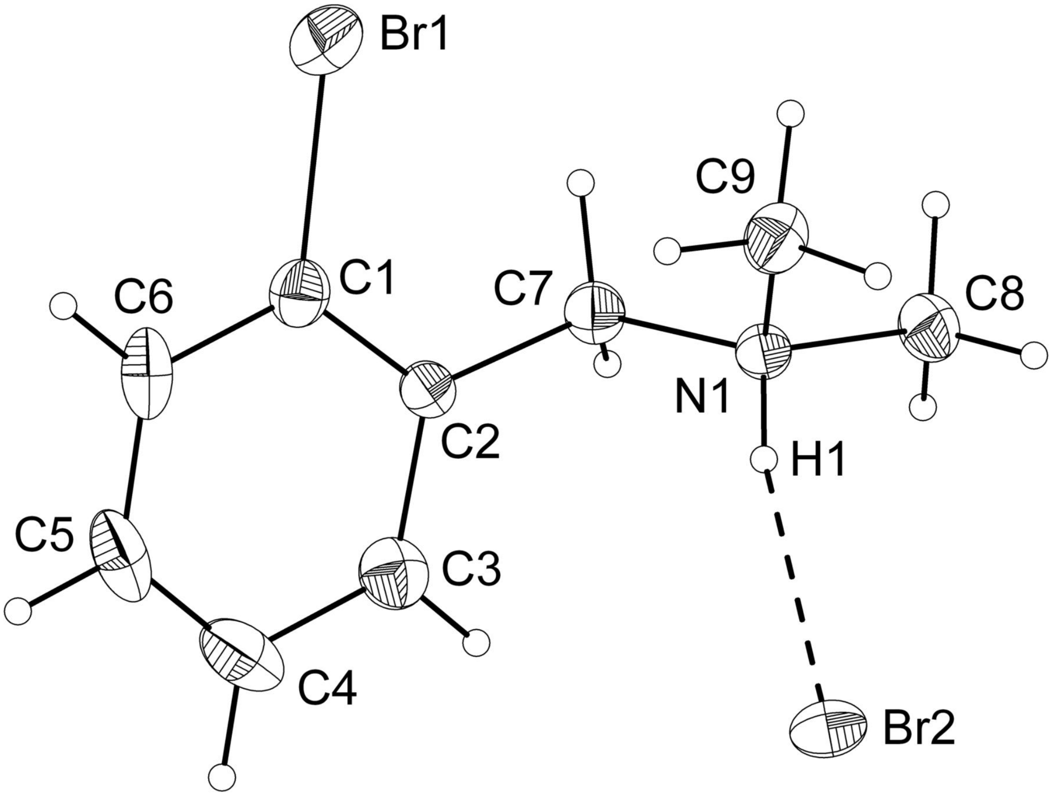


**Fig. 2**

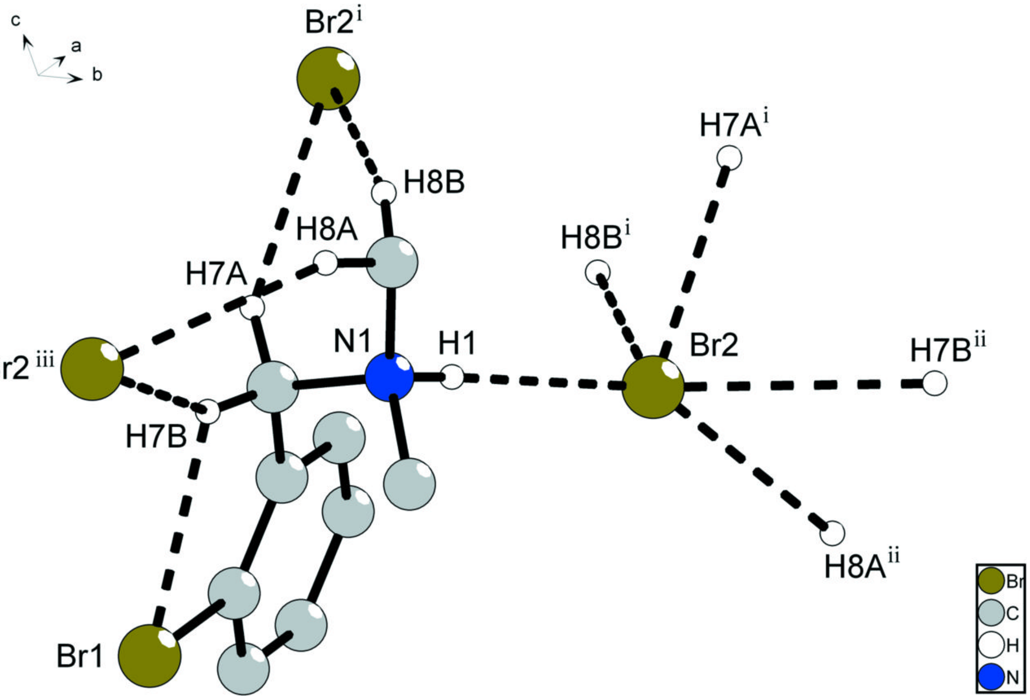

c
a
b
Br2$^{i}$
H7A$^{i}$
H8B
H8A
H8B$^{i}$
H7A
N1
H1
Br2
H7B$^{ii}$
Br2$^{iii}$
H7B
H8A$^{ii}$
Br1
Br
C
H
N

**Fig. 3**

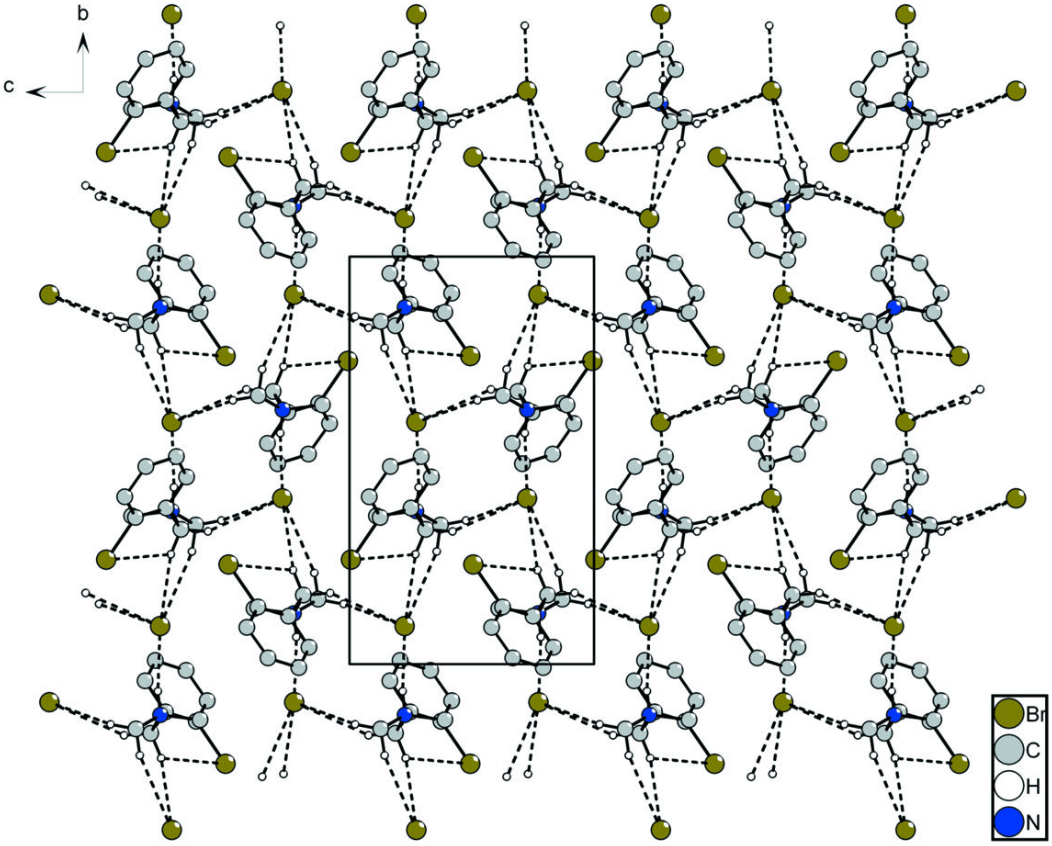


**Fig. 4**