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

ISSN 1600-5368

# Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-methylphenyl)stannate(IV)

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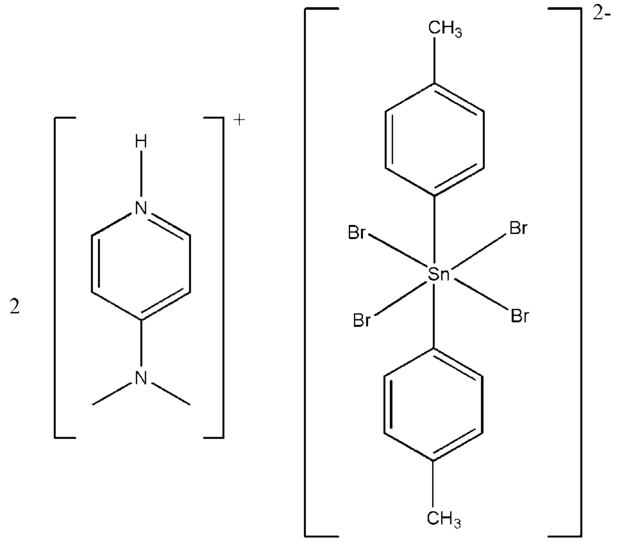
Received 8 June 2009; accepted 30 July 2009

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.018; $wR$ factor = 0.045; data-to-parameter ratio = 20.8.

In the title compound, $(C_7H_{11}N_2)_2[SnBr_4(C_7H_7)_2]$, the tetrabromidobis(4-methylphenyl)stannate(IV) anion possesses a centre of inversion located at the Sn$^{IV}$ atom. In the crystal structure, two inversion-related cations are linked to the anion *via* weak N—H···Br hydrogen bonds.

## Related literature

For related crystal structures, see Lo & Ng (2009); Koon *et al.* (2009); Yap *et al.* (2008).



## Experimental

### *Crystal data*

| | |
|---|---|
| $(C_7H_{11}N_2)_2[SnBr_4(C_7H_7)_2]$ | $V$ = 1555.64 (7) Å$^3$ |
| $M_r$ = 866.94 | $Z$ = 2 |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a$ = 10.2178 (3) Å | $\mu$ = 5.98 mm$^{-1}$ |
| $b$ = 10.4808 (3) Å | $T$ = 100 K |
| $c$ = 14.5833 (3) Å | 0.35 × 0.30 × 0.22 mm |
| $\beta$ = 95.063 (1)° | |

### *Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 11555 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | 3569 independent reflections |
| $T_{min}$ = 0.229, $T_{max}$ = 0.353 | 3225 reflections with $I > 2\sigma(I)$ |
| (expected range = 0.174–0.268) | $R_{int}$ = 0.019 |

### *Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.018 | 172 parameters |
| $wR(F^2)$ = 0.045 | H-atom parameters constrained |
| $S$ = 1.05 | $\Delta\rho_{max}$ = 0.44 e Å$^{-3}$ |
| 3569 reflections | $\Delta\rho_{min}$ = −0.45 e Å$^{-3}$ |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···Br1$^i$ | 0.88 | 2.75 | 3.448 (2) | 138 |
| N1—H1···Br2$^i$ | 0.88 | 2.94 | 3.517 (2) | 125 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the University of Malaya (grant No. PS072/2007C and PS320/2008C) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2842).

## References

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# supplementary materials

*Acta Cryst.* (2009). E**65**, m1040 [ **doi:10.1107/S1600536809030323** ]

# Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-methylphenyl)stannate(IV)

**S. M. Lee, K. M. Lo, H. Mohd Ali and W. T. Robinson**

## Comment

The molecular structure of the title compound is shown in Fig. 1.

## Experimental

Tetra(4-methylphenyl)tin (0.49 g, 1 mmol) and 4-dimethylaminopyridine hydrobromide perbromide (0.40 g, 1 mmol) were dissolved in absolute ethanol (25 ml) and refluxed for six hours. The solution was filtered and colourless crystals were isolated upon cooling to room temperature.

## Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95 to 0.98 Å) and were treated as riding on their parent carbon atoms, with *U*(H) set to 1.2–1.5 times *U*(C,*N*). N—H was refined and placed in the calculated position of N—H 0.88 ± 0.01 Å.

## Figures

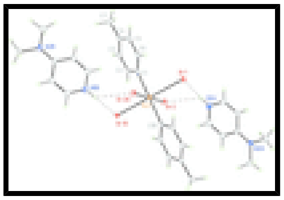

Fig. 1. The molecular structure of the title compound with 50% probability displacement ellipsoids and the atom numbering. Hydrogen atoms are drawn as spheres of arbitrary radius and dashed lines indicate hydrogen bonds (symmetry codes: (A) 1+x, -1+y, z and (B) 2-x, -y, -z).

## Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-methylphenyl)stannate(IV)

### *Crystal data*

| | |
|---|---|
| $(C_7H_{11}N_2)_2[SnBr_4(C_7H_7)_2]$ | $F_{000}$ = 844 |
| $M_r$ = 866.94 | $D_x$ = 1.851 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo *Kα* radiation, λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 6698 reflections |
| *a* = 10.2178 (3) Å | θ = 2.3–30.5° |
| *b* = 10.4808 (3) Å | μ = 5.98 mm$^{-1}$ |
| *c* = 14.5833 (3) Å | *T* = 100 K |
| β = 95.063 (1)° | Block, colourless |
| *V* = 1555.64 (7) Å$^3$ | 0.35 × 0.30 × 0.22 mm |
| *Z* = 2 | |

### *Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 3569 independent reflections |
| Radiation source: fine-focus sealed tube | 3225 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.019$ |
| $T = 100$ K | $\theta_{\text{max}} = 27.5°$ |
| ω scans | $\theta_{\text{min}} = 2.3°$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -13\rightarrow10$ |
| $T_{\text{min}} = 0.229$, $T_{\text{max}} = 0.353$ | $k = -13\rightarrow8$ |
| 11555 measured reflections | $l = -18\rightarrow18$ |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.018$ | H-atom parameters constrained |
| $wR(F^2) = 0.045$ | $w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 0.5727P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.05$ | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 3569 reflections | $\Delta\rho_{\text{max}} = 0.44$ e Å$^{-3}$ |
| 172 parameters | $\Delta\rho_{\text{min}} = -0.45$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | 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 $F^2 > \sigma(F^2)$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{\text{iso}}$*/$U_{\text{eq}}$ |
|---|---|---|---|---|
| Sn1 | 1.0000 | 0.0000 | 0.0000 | 0.01042 (5) |
| Br1 | 0.951470 (19) | −0.180492 (17) | 0.131083 (12) | 0.01383 (5) |
| Br2 | 1.261950 (18) | −0.018292 (18) | 0.055861 (14) | 0.01597 (6) |
| N1 | 0.24150 (18) | 0.65685 (17) | 0.11335 (13) | 0.0237 (4) |
| H1 | 0.2028 | 0.7281 | 0.1280 | 0.028* |

| | | | | |
|---|---|---|---|---|
| N2 | 0.43055 (16) | 0.33066 (16) | 0.04249 (11) | 0.0166 (3) |
| C1 | 0.98097 (18) | 0.15055 (17) | 0.09731 (12) | 0.0106 (3) |
| C2 | 1.06827 (19) | 0.25260 (18) | 0.10370 (13) | 0.0140 (4) |
| H2 | 1.1417 | 0.2527 | 0.0680 | 0.017* |
| C3 | 1.04872 (19) | 0.35431 (19) | 0.16189 (13) | 0.0158 (4) |
| H3 | 1.1089 | 0.4236 | 0.1651 | 0.019* |
| C4 | 0.94235 (19) | 0.35675 (19) | 0.21573 (13) | 0.0148 (4) |
| C5 | 0.85693 (19) | 0.25278 (19) | 0.21028 (13) | 0.0142 (4) |
| H5 | 0.7847 | 0.2514 | 0.2471 | 0.017* |
| C6 | 0.87603 (18) | 0.15088 (18) | 0.15165 (12) | 0.0136 (4) |
| H6 | 0.8167 | 0.0809 | 0.1488 | 0.016* |
| C7 | 0.9200 (2) | 0.46954 (19) | 0.27623 (15) | 0.0204 (4) |
| H7A | 0.8970 | 0.5443 | 0.2377 | 0.031* |
| H7B | 1.0004 | 0.4873 | 0.3160 | 0.031* |
| H7C | 0.8482 | 0.4506 | 0.3144 | 0.031* |
| C8 | 0.2184 (2) | 0.6093 (2) | 0.02768 (15) | 0.0249 (5) |
| H8 | 0.1593 | 0.6526 | −0.0157 | 0.030* |
| C9 | 0.2777 (2) | 0.5010 (2) | 0.00204 (15) | 0.0201 (4) |
| H9 | 0.2596 | 0.4687 | −0.0586 | 0.024* |
| C10 | 0.36716 (18) | 0.43551 (18) | 0.06587 (13) | 0.0131 (4) |
| C11 | 0.3857 (2) | 0.48772 (18) | 0.15624 (14) | 0.0171 (4) |
| H11 | 0.4421 | 0.4461 | 0.2022 | 0.021* |
| C12 | 0.3229 (2) | 0.5969 (2) | 0.17697 (14) | 0.0202 (4) |
| H12 | 0.3367 | 0.6314 | 0.2373 | 0.024* |
| C13 | 0.4097 (2) | 0.2765 (2) | −0.05001 (14) | 0.0208 (4) |
| H13A | 0.3200 | 0.2431 | −0.0599 | 0.031* |
| H13B | 0.4726 | 0.2072 | −0.0565 | 0.031* |
| H13C | 0.4225 | 0.3430 | −0.0956 | 0.031* |
| C14 | 0.5171 (2) | 0.2619 (2) | 0.11039 (15) | 0.0249 (5) |
| H14A | 0.5837 | 0.3204 | 0.1387 | 0.037* |
| H14B | 0.5603 | 0.1920 | 0.0801 | 0.037* |
| H14C | 0.4653 | 0.2270 | 0.1580 | 0.037* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn1 | 0.01059 (9) | 0.00929 (9) | 0.01105 (9) | 0.00024 (6) | −0.00081 (6) | −0.00130 (6) |
| Br1 | 0.01585 (10) | 0.01282 (10) | 0.01285 (9) | −0.00079 (7) | 0.00140 (7) | 0.00179 (7) |
| Br2 | 0.01007 (10) | 0.01519 (10) | 0.02186 (11) | 0.00061 (7) | −0.00312 (7) | −0.00193 (7) |
| N1 | 0.0260 (10) | 0.0161 (9) | 0.0291 (10) | 0.0075 (7) | 0.0021 (8) | 0.0008 (7) |
| N2 | 0.0155 (8) | 0.0160 (8) | 0.0179 (8) | 0.0010 (7) | −0.0002 (6) | 0.0011 (7) |
| C1 | 0.0124 (9) | 0.0099 (8) | 0.0091 (8) | 0.0006 (7) | −0.0018 (7) | −0.0002 (7) |
| C2 | 0.0120 (9) | 0.0134 (9) | 0.0165 (9) | −0.0010 (7) | 0.0013 (7) | −0.0003 (7) |
| C3 | 0.0139 (9) | 0.0119 (9) | 0.0210 (10) | −0.0036 (7) | −0.0009 (7) | −0.0015 (7) |
| C4 | 0.0157 (9) | 0.0137 (9) | 0.0143 (9) | 0.0041 (8) | −0.0027 (7) | −0.0022 (7) |
| C5 | 0.0119 (9) | 0.0185 (10) | 0.0124 (9) | 0.0022 (7) | 0.0024 (7) | 0.0002 (7) |
| C6 | 0.0126 (9) | 0.0143 (9) | 0.0136 (9) | −0.0019 (7) | −0.0006 (7) | 0.0005 (7) |
| C7 | 0.0218 (11) | 0.0158 (10) | 0.0232 (10) | 0.0042 (8) | 0.0005 (8) | −0.0068 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8 | 0.0252 (11) | 0.0265 (12) | 0.0225 (11) | 0.0073 (9) | −0.0016 (9) | 0.0083 (9) |
| C9 | 0.0193 (11) | 0.0246 (11) | 0.0157 (10) | 0.0023 (8) | −0.0021 (8) | 0.0035 (8) |
| C10 | 0.0110 (9) | 0.0119 (9) | 0.0165 (9) | −0.0037 (7) | 0.0011 (7) | 0.0020 (7) |
| C11 | 0.0153 (10) | 0.0175 (10) | 0.0177 (10) | −0.0019 (8) | −0.0035 (8) | 0.0019 (8) |
| C12 | 0.0208 (11) | 0.0179 (10) | 0.0214 (10) | −0.0019 (8) | −0.0012 (8) | −0.0016 (8) |
| C13 | 0.0203 (11) | 0.0228 (11) | 0.0194 (10) | −0.0016 (9) | 0.0025 (8) | −0.0038 (8) |
| C14 | 0.0275 (12) | 0.0205 (11) | 0.0259 (11) | 0.0108 (9) | −0.0016 (9) | 0.0030 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Sn1—C1$^{i}$ | 2.1424 (18) | C5—C6 | 1.393 (3) |
| Sn1—C1 | 2.1424 (18) | C5—H5 | 0.9500 |
| Sn1—Br2$^{i}$ | 2.7349 (2) | C6—H6 | 0.9500 |
| Sn1—Br2 | 2.7349 (2) | C7—H7A | 0.9800 |
| Sn1—Br1 | 2.76515 (18) | C7—H7B | 0.9800 |
| Sn1—Br1$^{i}$ | 2.76515 (18) | C7—H7C | 0.9800 |
| N1—C12 | 1.346 (3) | C8—C9 | 1.356 (3) |
| N1—C8 | 1.346 (3) | C8—H8 | 0.9500 |
| N1—H1 | 0.8800 | C9—C10 | 1.422 (3) |
| N2—C10 | 1.335 (2) | C9—H9 | 0.9500 |
| N2—C14 | 1.459 (3) | C10—C11 | 1.424 (3) |
| N2—C13 | 1.462 (3) | C11—C12 | 1.359 (3) |
| C1—C6 | 1.388 (3) | C11—H11 | 0.9500 |
| C1—C2 | 1.391 (3) | C12—H12 | 0.9500 |
| C2—C3 | 1.388 (3) | C13—H13A | 0.9800 |
| C2—H2 | 0.9500 | C13—H13B | 0.9800 |
| C3—C4 | 1.396 (3) | C13—H13C | 0.9800 |
| C3—H3 | 0.9500 | C14—H14A | 0.9800 |
| C4—C5 | 1.394 (3) | C14—H14B | 0.9800 |
| C4—C7 | 1.504 (3) | C14—H14C | 0.9800 |
| C1$^{i}$—Sn1—C1 | 180.00 (13) | C1—C6—C5 | 120.63 (18) |
| C1$^{i}$—Sn1—Br2$^{i}$ | 89.88 (5) | C1—C6—H6 | 119.7 |
| C1—Sn1—Br2$^{i}$ | 90.12 (5) | C5—C6—H6 | 119.7 |
| C1$^{i}$—Sn1—Br2 | 90.12 (5) | C4—C7—H7A | 109.5 |
| C1—Sn1—Br2 | 89.88 (5) | C4—C7—H7B | 109.5 |
| Br2$^{i}$—Sn1—Br2 | 180.000 (12) | H7A—C7—H7B | 109.5 |
| C1$^{i}$—Sn1—Br1 | 89.22 (5) | C4—C7—H7C | 109.5 |
| C1—Sn1—Br1 | 90.78 (5) | H7A—C7—H7C | 109.5 |
| Br2$^{i}$—Sn1—Br1 | 91.340 (6) | H7B—C7—H7C | 109.5 |
| Br2—Sn1—Br1 | 88.660 (6) | N1—C8—C9 | 121.3 (2) |
| C1$^{i}$—Sn1—Br1$^{i}$ | 90.78 (5) | N1—C8—H8 | 119.3 |
| C1—Sn1—Br1$^{i}$ | 89.22 (5) | C9—C8—H8 | 119.3 |
| Br2$^{i}$—Sn1—Br1$^{i}$ | 88.660 (6) | C8—C9—C10 | 120.1 (2) |
| Br2—Sn1—Br1$^{i}$ | 91.340 (6) | C8—C9—H9 | 120.0 |
| Br1—Sn1—Br1$^{i}$ | 180.000 (10) | C10—C9—H9 | 120.0 |
| C12—N1—C8 | 120.94 (19) | N2—C10—C9 | 121.88 (18) |

| | | | |
|---|---|---|---|
| C12—N1—H1 | 119.5 | N2—C10—C11 | 121.65 (18) |
| C8—N1—H1 | 119.5 | C9—C10—C11 | 116.47 (18) |
| C10—N2—C14 | 120.81 (17) | C12—C11—C10 | 120.16 (19) |
| C10—N2—C13 | 121.38 (17) | C12—C11—H11 | 119.9 |
| C14—N2—C13 | 117.71 (17) | C10—C11—H11 | 119.9 |
| C6—C1—C2 | 118.88 (17) | N1—C12—C11 | 121.0 (2) |
| C6—C1—Sn1 | 119.98 (13) | N1—C12—H12 | 119.5 |
| C2—C1—Sn1 | 121.04 (13) | C11—C12—H12 | 119.5 |
| C3—C2—C1 | 120.37 (17) | N2—C13—H13A | 109.5 |
| C3—C2—H2 | 119.8 | N2—C13—H13B | 109.5 |
| C1—C2—H2 | 119.8 | H13A—C13—H13B | 109.5 |
| C2—C3—C4 | 121.28 (18) | N2—C13—H13C | 109.5 |
| C2—C3—H3 | 119.4 | H13A—C13—H13C | 109.5 |
| C4—C3—H3 | 119.4 | H13B—C13—H13C | 109.5 |
| C5—C4—C3 | 117.91 (17) | N2—C14—H14A | 109.5 |
| C5—C4—C7 | 121.42 (17) | N2—C14—H14B | 109.5 |
| C3—C4—C7 | 120.66 (18) | H14A—C14—H14B | 109.5 |
| C6—C5—C4 | 120.90 (17) | N2—C14—H14C | 109.5 |
| C6—C5—H5 | 119.5 | H14A—C14—H14C | 109.5 |
| C4—C5—H5 | 119.5 | H14B—C14—H14C | 109.5 |

| | | | |
|---|---|---|---|
| C1$^{i}$—Sn1—C1—C6 | 0.00 (18) | C7—C4—C5—C6 | 177.77 (18) |
| Br2$^{i}$—Sn1—C1—C6 | 44.67 (14) | C2—C1—C6—C5 | 1.2 (3) |
| Br2—Sn1—C1—C6 | −135.33 (14) | Sn1—C1—C6—C5 | −175.23 (14) |
| Br1—Sn1—C1—C6 | −46.67 (14) | C4—C5—C6—C1 | 0.1 (3) |
| Br1$^{i}$—Sn1—C1—C6 | 133.33 (14) | C12—N1—C8—C9 | −1.1 (3) |
| C1$^{i}$—Sn1—C1—C2 | 0.00 (7) | N1—C8—C9—C10 | −0.4 (3) |
| Br2$^{i}$—Sn1—C1—C2 | −131.72 (15) | C14—N2—C10—C9 | −177.10 (19) |
| Br2—Sn1—C1—C2 | 48.28 (15) | C13—N2—C10—C9 | −0.9 (3) |
| Br1—Sn1—C1—C2 | 136.94 (15) | C14—N2—C10—C11 | 2.9 (3) |
| Br1$^{i}$—Sn1—C1—C2 | −43.06 (15) | C13—N2—C10—C11 | 179.12 (18) |
| C6—C1—C2—C3 | −1.5 (3) | C8—C9—C10—N2 | −178.0 (2) |
| Sn1—C1—C2—C3 | 174.92 (14) | C8—C9—C10—C11 | 2.0 (3) |
| C1—C2—C3—C4 | 0.5 (3) | N2—C10—C11—C12 | 177.86 (18) |
| C2—C3—C4—C5 | 0.9 (3) | C9—C10—C11—C12 | −2.1 (3) |
| C2—C3—C4—C7 | −178.05 (19) | C8—N1—C12—C11 | 1.0 (3) |
| C3—C4—C5—C6 | −1.1 (3) | C10—C11—C12—N1 | 0.7 (3) |

Symmetry codes: (i) −*x*+2, −*y*, −*z*.

### *Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Br1$^{ii}$ | 0.88 | 2.75 | 3.448 (2) | 138 |
| N1—H1···Br2$^{ii}$ | 0.88 | 2.94 | 3.517 (2) | 125 |

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

Fig. 1