

Bis(2-bromopyridinium) hexabromido-stannate(IV)

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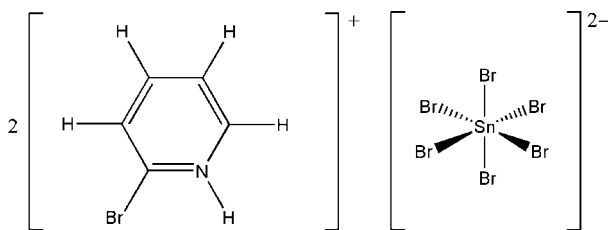
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.020$ Å; R factor = 0.068; wR factor = 0.178; data-to-parameter ratio = 27.0.

The asymmetric unit of the title compound, $(\text{C}_5\text{H}_5\text{BrN})_2\text{[SnBr}_6\text{]}$, contains one cation and one half-anion. The $[\text{SnBr}_6]^{2-}$ anion is located on an inversion center and forms a quasi-regular octahedral arrangement. The crystal structure consists of two-dimensional supramolecular layers assembled via hydrogen-bonding interactions of $\text{N}-\text{H}\cdots\text{Br}-\text{Sn}$ [$D\cdots A = 3.375$ (13)– 3.562 (13) Å and $D-\text{H}\cdots A = 127$ – 142° , along with $\text{C}-\text{Br}\cdots\text{Br}$ synthons [3.667 (2) and 3.778 (3) Å]. These layers are parallel to the bc plane and built up from anions interacting extensively with the six surrounding cations.

Related literature

The title salt is isomorphous with the Te analogue (Fernandes *et al.*, 2004). For related literature, see: Al-Far & Ali (2007); Ali, Al-Far & Al-Sou'od (2007); Ali & Al-Far (2007); Ali, Al-Far & Ng (2007); Allen *et al.* (1987); Aruta *et al.* (2005); Hill (1998); Kagan *et al.* (1999); Knutson *et al.* (2005); Raptopoulou *et al.* (2002); Tudela & Khan (1991); Willey *et al.* (1998).



Experimental

Crystal data

 $(\text{C}_5\text{H}_5\text{BrN})_2[\text{SnBr}_6]$
 $M_r = 916.12$
 Triclinic, $P\bar{1}$
 $a = 7.4037$ (15) Å
 $b = 8.3393$ (17) Å
 $c = 9.4302$ (19) Å

 $\alpha = 73.14$ (3)°
 $\beta = 67.98$ (3)°
 $\gamma = 82.44$ (3)°
 $V = 516.4$ (2) Å³
 $Z = 1$
 Mo $K\alpha$ radiation

 $\mu = 16.71$ mm⁻¹
 $T = 293$ (2) K

 $0.16 \times 0.13 \times 0.08$ mm

Data collection

 Bruker–Siemens SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2007)
 $T_{\min} = 0.058$, $T_{\max} = 0.261$

 2266 measured reflections
 1807 independent reflections
 1308 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.091$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.178$
 $S = 1.02$
 1807 reflections

 67 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 3.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.87$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

| | | | |
|---------------------------|-------------|--------------------------|-------------|
| Sn1—Br3 | 2.5939 (15) | Sn1—Br4 | 2.6174 (17) |
| Sn1—Br1 | 2.6027 (15) | | |
| Br3—Sn1—Br1 | 89.06 (5) | Br1—Sn1—Br4 ⁱ | 90.21 (5) |
| Br3 ⁱ —Sn1—Br1 | 90.94 (5) | Br3—Sn1—Br4 | 90.57 (6) |
| Br3—Sn1—Br4 ⁱ | 89.43 (6) | Br1—Sn1—Br4 | 89.79 (5) |

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

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| N1—H1 ⁱⁱ ···Br4 ⁱⁱ | 0.86 | 2.65 | 3.375 (13) | 142 |
| N1—H1 ⁱⁱⁱ ···Br1 ⁱⁱⁱ | 0.86 | 2.98 | 3.562 (13) | 127 |

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

Data collection: SMART (Bruker, 2006); cell refinement: SAINT-Plus (Bruker, 2006); data reduction: SAINT-Plus; program(s) used to solve structure: XS in SHELXTL (Sheldrick, 2008); program(s) used to refine structure: XL in SHELXTL; molecular graphics: XP in SHELXTL; software used to prepare material for publication: XCIF in SHELXTL.

Al al-Bayt University and Al-Balqa'a Applied University are thanked for supporting this work

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2139).

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supplementary materials

Acta Cryst. (2008). E64, m749-m750 [doi:10.1107/S1600536808012129]

Bis(2-bromopyridinium) hexabromidostannate(IV)

B. F. Ali, R. H. Al-Far and S. F. Haddad

Comment

Noncovalent interactions play an important role in organizing structural units in both natural and artificial systems. Hybrid organic-inorganic compounds are of great interest owing to their ionic, electrical, magnetic and optical properties (Hill, 1998; Kagan *et al.*, 1999; Raptopoulou *et al.*, 2002). Tin metal-halo based hybrids are of particular interest as being materials with interesting optical and magnetic properties (Aruta *et al.*, 2005; Knutson *et al.*, 2005; Kagan *et al.*, 1999). We are currently carrying out studies about lattice including different types of intermolecular interactions (aryl \cdots aryl, $X\cdots X$, $X\cdots$ aryl and $X\cdots H$). Within our research of hybrid compounds containing tin metal (Al-Far & Ali 2007; Ali, Al-Far & Al-Sou'od, 2007; Ali & Al-Far, 2007; Ali, Al-Far & Ng, 2007), the crystal structure of the title salt, (I), has been investigated.

The asymmetric unit of (I) contains one cation and one-half anion (Fig. 1). The whole $(2\text{-Br}-\text{C}_5\text{H}_5\text{N})_2[\text{SnBr}_6]$ geometry is generated through an inversion center with tin being lying on the special crystallographic position of $(1/2, 1/2, 0)$. The $(\text{SnBr}_6)^{2-}$ anion forms a quasi-octahedral geometry (Table 1), with the Sn—Br bond lengths are almost invariant. These lengths are in accordance with tin-bromide distances reported for $(\text{SnBr}_6)^{2-}$ anion containing compounds (Willey *et al.*, 1998; Tudela & Khan 1991; Al-Far & Ali 2007; Ali, Al-Far & Al-Sou'od, 2007; Ali & Al-Far, 2007; Ali, Al-Far & Ng, 2007). Bond lengths and angles within the cation are as expected (Allen *et al.*, 1987).

The packing of the structure (Fig. 2) can be described as layers of alternating anions and cations parallel to bc plane. In these layers each $(\text{SnBr}_6)^{2-}$ anion is interacting with six cations *via* two N—H \cdots Br interactions (Table 2) and the symmetry related ones along with two Br \cdots Br interactions and symmetry related ones [Br2 \cdots Br4 and Br2 \cdots Br1 of 3.6666 (23) and 3.7779 (29) Å, respectively; Fig. 2].

The N—H \cdots N interactions along with C—Br \cdots Br synthons are potential building blocks for this stable supramolecular lattice. The stability of this lattice is evident in the isostructurality with the reported Te analogue (Fernandes *et al.*, 2004).

Experimental

Warm solution of Sn metal (1.0 mmol) dissolved in absolute ethanol (10 ml) and HBr (60%, 5 ml), was added dropwise to a stirred hot solution of 2-bromopyridine (2 mmol) dissolved in ethanol (10 ml). The mixture was then treated with liquid Br₂ (2 ml) and refluxed for 3/2 h. The resulting mixture was then filtered off, and allowed to stand undisturbed at room temperature. The salt crystallized over 1 d as nice yellow block crystals (yield: 83%).

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Figures

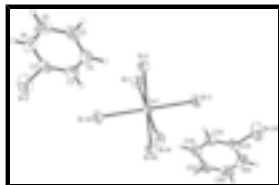


Fig. 1. A view of the asymmetric unit of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

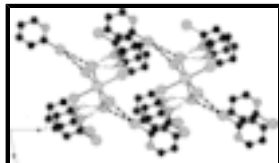


Fig. 2. A packing diagram of (I). Hydrogen bonds (dashed lines) and Br...Br interactions (thick dashed lines) are shown for $(\text{SnBr}_6)^{2-}$ anions and six surrounding cations.

Bis(2-bromopyridinium) hexabromidostannate(IV)

Crystal data

$(\text{C}_5\text{H}_5\text{BrN})_2[\text{SnBr}_6]$

$M_r = 916.12$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4037(15) \text{ \AA}$

$b = 8.3393(17) \text{ \AA}$

$c = 9.4302(19) \text{ \AA}$

$\alpha = 73.14(3)^\circ$

$\beta = 67.98(3)^\circ$

$\gamma = 82.44(3)^\circ$

$V = 516.4(2) \text{ \AA}^3$

$Z = 1$

$F_{000} = 414$

$D_x = 2.946 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 255 reflections

$\theta = 2.1\text{--}27.7^\circ$

$\mu = 16.71 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Block, yellow

$0.16 \times 0.13 \times 0.08 \text{ mm}$

Data collection

Bruker-Siemens SMART APEX diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: $8.3 \text{ pixels mm}^{-1}$

$T = 293(2) \text{ K}$

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\min} = 0.058$, $T_{\max} = 0.261$

2266 measured reflections

1807 independent reflections

1308 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.091$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.4^\circ$

$h = -1 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -10 \rightarrow 11$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.068$$

$$wR(F^2) = 0.178$$

$$S = 1.02$$

1807 reflections

67 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1076P)^2 + 1.002P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 3.31 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.87 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| Sn1 | 0.5000 | 0.5000 | 0.0000 | 0.0271 (4) |
| Br4 | 0.80819 (19) | 0.51691 (17) | -0.25738 (17) | 0.0365 (4) |
| Br3 | 0.5601 (2) | 0.17999 (16) | 0.09614 (18) | 0.0394 (4) |
| Br1 | 0.2879 (2) | 0.43877 (19) | -0.14375 (19) | 0.0426 (4) |
| Br2 | 0.6761 (3) | 0.2489 (2) | -0.4484 (2) | 0.0674 (6) |
| C2 | 0.7936 (19) | 0.1051 (18) | -0.5793 (18) | 0.038 (3)* |
| N1 | 0.9316 (17) | 0.1687 (17) | -0.7225 (17) | 0.049 (3)* |
| H1 | 0.9575 | 0.2732 | -0.7499 | 0.059* |
| C3 | 0.751 (2) | -0.0578 (18) | -0.5321 (19) | 0.042 (3)* |
| H3 | 0.6604 | -0.1039 | -0.4324 | 0.051* |
| C4 | 0.845 (2) | -0.155 (2) | -0.6371 (19) | 0.047 (4)* |
| H4 | 0.8119 | -0.2665 | -0.6094 | 0.057* |
| C5 | 0.990 (2) | -0.090 (2) | -0.784 (2) | 0.051 (4)* |
| H5 | 1.0572 | -0.1560 | -0.8526 | 0.061* |
| C6 | 1.028 (3) | 0.077 (2) | -0.822 (2) | 0.060 (5)* |
| H6 | 1.1225 | 0.1254 | -0.9183 | 0.072* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| Sn1 | 0.0304 (6) | 0.0249 (6) | 0.0213 (7) | 0.0021 (5) | -0.0058 (5) | -0.0048 (5) |
| Br4 | 0.0387 (7) | 0.0322 (7) | 0.0294 (8) | -0.0003 (5) | -0.0011 (6) | -0.0094 (6) |

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|--------------|-------------|
| Br3 | 0.0462 (8) | 0.0258 (7) | 0.0340 (9) | 0.0066 (6) | -0.0067 (6) | -0.0034 (6) |
| Br1 | 0.0491 (8) | 0.0446 (8) | 0.0391 (9) | -0.0002 (6) | -0.0226 (7) | -0.0094 (7) |
| Br2 | 0.1089 (15) | 0.0522 (10) | 0.0491 (11) | 0.0059 (10) | -0.0295 (11) | -0.0268 (9) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--|-------------|-------------|------------|
| Sn1—Br3 | 2.5939 (15) | N1—C6 | 1.32 (2) |
| Sn1—Br3 ⁱ | 2.5939 (15) | N1—H1 | 0.8600 |
| Sn1—Br1 | 2.6027 (15) | C3—C4 | 1.39 (2) |
| Sn1—Br1 ⁱ | 2.6027 (15) | C3—H3 | 0.9300 |
| Sn1—Br4 ⁱ | 2.6174 (17) | C4—C5 | 1.40 (2) |
| Sn1—Br4 | 2.6174 (17) | C4—H4 | 0.9300 |
| Br2—C2 | 1.870 (15) | C5—C6 | 1.37 (2) |
| C2—C3 | 1.34 (2) | C5—H5 | 0.9300 |
| C2—N1 | 1.357 (19) | C6—H6 | 0.9300 |
| Br3—Sn1—Br3 ⁱ | 180.0 | N1—C2—Br2 | 117.9 (11) |
| Br3—Sn1—Br1 | 89.06 (5) | C6—N1—C2 | 122.7 (14) |
| Br3 ⁱ —Sn1—Br1 | 90.94 (5) | C6—N1—H1 | 118.6 |
| Br3—Sn1—Br1 ⁱ | 90.94 (5) | C2—N1—H1 | 118.6 |
| Br3 ⁱ —Sn1—Br1 ⁱ | 89.06 (5) | C2—C3—C4 | 117.8 (15) |
| Br1—Sn1—Br1 ⁱ | 180.00 (5) | C2—C3—H3 | 121.1 |
| Br3—Sn1—Br4 ⁱ | 89.43 (6) | C4—C3—H3 | 121.1 |
| Br3 ⁱ —Sn1—Br4 ⁱ | 90.57 (6) | C3—C4—C5 | 121.7 (15) |
| Br1—Sn1—Br4 ⁱ | 90.21 (5) | C3—C4—H4 | 119.1 |
| Br1 ⁱ —Sn1—Br4 ⁱ | 89.79 (5) | C5—C4—H4 | 119.1 |
| Br3—Sn1—Br4 | 90.57 (6) | C6—C5—C4 | 116.8 (17) |
| Br3 ⁱ —Sn1—Br4 | 89.43 (6) | C6—C5—H5 | 121.6 |
| Br1—Sn1—Br4 | 89.79 (5) | C4—C5—H5 | 121.6 |
| Br1 ⁱ —Sn1—Br4 | 90.21 (5) | N1—C6—C5 | 120.6 (17) |
| Br4 ⁱ —Sn1—Br4 | 180.0 | N1—C6—H6 | 119.7 |
| C3—C2—N1 | 120.3 (15) | C5—C6—H6 | 119.7 |
| C3—C2—Br2 | 121.8 (12) | | |
| C3—C2—N1—C6 | 1(2) | C2—C3—C4—C5 | 4(2) |
| Br2—C2—N1—C6 | 177.7 (12) | C3—C4—C5—C6 | -3(2) |
| N1—C2—C3—C4 | -3(2) | C2—N1—C6—C5 | 1(3) |
| Br2—C2—C3—C4 | -179.9 (11) | C4—C5—C6—N1 | 0(3) |

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

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| N1—H1 \cdots Br4 ⁱⁱ | 0.86 | 2.65 | 3.375 (13) | 142 |
| N1—H1 \cdots Br1 ⁱⁱⁱ | 0.86 | 2.98 | 3.562 (13) | 127 |

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

Fig. 1

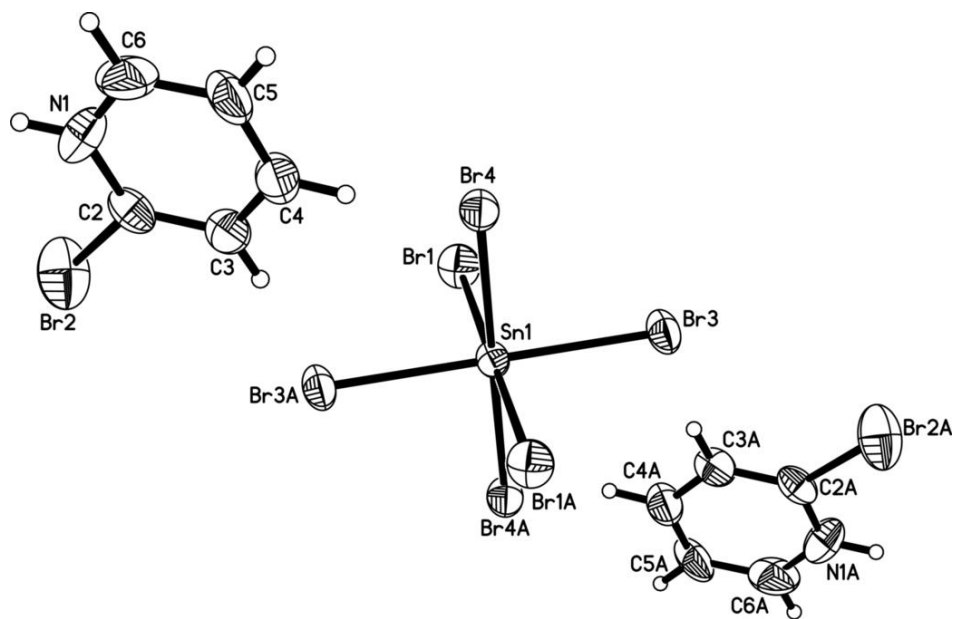


Fig. 2

