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# Bis[4-(dimethylamino)pyridinium] tetrabromidodiphenylstannate(IV)

**Quai Ling Yap, Kong Mun Lo and Seik Weng Ng***

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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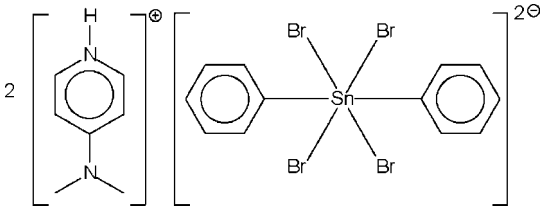
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.024; $wR$ factor = 0.052; data-to-parameter ratio = 20.1.

The $Sn^{IV}$ atom of the stannate anion in the title salt, $(C_7H_{11}N_2)_2[SnBr_4(C_6H_5)_2]$, lies on a center of inversion in a tetragonally compressed octahedron. The two independent Br atoms in the anion are hydrogen-bond acceptors for the same cation.

## Related literature

For the structure of dipyridinium tetrabromidostannate(II), see: Tuleda & Khan (1991).




## Experimental

### Crystal data

$(C_7H_{11}N_2)_2[SnBr_4(C_6H_5)_2]$
$M_r$ = 838.89
Monoclinic, $P2_1/n$
$a$ = 10.7803 (2) Å
$b$ = 9.3847 (2) Å
$c$ = 14.4068 (4) Å
$\beta$ = 94.126 (2)°
$V$ = 1453.76 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 6.40 mm$^{-1}$
$T$ = 100 (2) K
0.24 × 0.18 × 0.12 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.386, $T_{max}$ = 0.514 (expected range = 0.348–0.464)
11853 measured reflections
3334 independent reflections
2688 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.051
$S$ = 0.99
3334 reflections
166 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.42 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Sn1—C1 | 2.143 (3) | Sn1—Br2 | 2.7470 (3) |
| Sn1—Br1 | 2.7395 (2) | | |
| C1—Sn1—Br1 | 90.53 (7) | C1—Sn1—Br2$^{i}$ | 90.36 (7) |
| C1—Sn1—Br1$^{i}$ | 89.47 (7) | Br1—Sn1—Br2 | 88.981 (8) |
| C1—Sn1—Br2 | 89.64 (7) | Br1—Sn1—Br2$^{i}$ | 91.019 (8) |

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2N···Br1 | 0.88 (1) | 2.79 (3) | 3.385 (3) | 126 (3) |
| N2—H2N···Br2 | 0.88 (1) | 2.81 (3) | 3.485 (3) | 135 (3) |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2264).

## References

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# supplementary materials

*Acta Cryst.* (2008). E**64**, m696 [ **doi:10.1107/S1600536808010830** ]

# Bis[4-(dimethylamino)pyridinium] tetrabromidodiphenylstannate(IV)

**Q. L. Yap, K. M. Lo and S. W. Ng**

## Comment

Bis[4-(dimethylamino)pyridinium] tetrabromidodiphenylstannate(IV), (I) (Fig. 1 and Table 1) was the product of the cleavage of the mixed alkyl/triarylstannate, cyclopentyltriphenyltin, by 4-dimethylaminopyridine hydrobromide perbromide. The stannate has the tin atom in a tetragonally compressed octahedral $Br_4C_2$ environment. The anion has also been reported as the centrosymmetric pyridinium salt: Sn–Br = 2.7592 (3), 2.7737 (3) and Sn–C 2.158 (3) Å (Tuleda & Khan, 1991). Connections between ions are of the type N-H···Br (Table 2) so that each independent pair of bromide atoms are linked to the same cation.

## Experimental

Cyclopentyltriphenyltin (1.36 g, 3 mmol) and 4-dimethylaminopyridine hydrobromide perbromide (1.1 g, 3 mmol) were heated in chloroform (100 ml) for 3 h. The filtered solution when allowed to evaporate yielded large yellow crystals, m.p. 470–473 K.

## Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2 to 1.5$U_{eq}$(C). The ammonium H atom was refined with a distance restraint of N–H 0.88±0.01 Å; its displacement parameter was freely refined.

## Figures

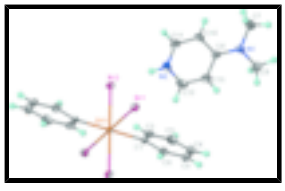

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of $[C_7H_{11}N]_2$ $[SnBr_4(C_6H_5)_2]$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

## Bis[4-(dimethylamino)pyridinium] tetrabromidodiphenylstannate(IV)

*Crystal data*

| | |
|---|---|
| $(C_7H_{11}N_2)_2[SnBr_4(C_6H_5)_2]$ | $F_{000}$ = 812 |
| $M_r$ = 838.89 | $D_x$ = 1.916 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 3449 reflections |

| | |
|---|---|
| $a$ = 10.7803 (2) Å | θ = 2.3–28.3º |
| $b$ = 9.3847 (2) Å | μ = 6.40 mm$^{-1}$ |
| $c$ = 14.4068 (4) Å | $T$ = 100 (2) K |
| β = 94.126 (2)º | Block, colorless |
| $V$ = 1453.76 (6) Å$^3$ | 0.24 × 0.18 × 0.12 mm |
| $Z$ = 2 | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 3334 independent reflections |
| Radiation source: fine-focus sealed tube | 2688 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.035 |
| $T$ = 100(2) K | $\theta_{max}$ = 27.5º |
| ω scans | $\theta_{min}$ = 2.3º |
| Absorption correction: Multi-scan (SADABS; Sheldrick, 1996) | $h$ = −14→14 |
| $T_{min}$ = 0.386, $T_{max}$ = 0.514 | $k$ = −12→12 |
| 11853 measured reflections | $l$ = −18→14 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.024 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.051 | $w = 1/[\sigma^2(F_o^2) + (0.0228P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 0.99 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 3334 reflections | $\Delta\rho_{max}$ = 0.47 e Å$^{-3}$ |
| 166 parameters | $\Delta\rho_{min}$ = −0.42 e Å$^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Sn1 | 0.5000 | 0.5000 | 0.5000 | 0.01159 (7) |
| Br1 | 0.25635 (2) | 0.50711 (3) | 0.43147 (2) | 0.01591 (7) |
| Br2 | 0.55014 (2) | 0.71661 (3) | 0.38004 (2) | 0.01549 (7) |
| N1 | 0.05057 (19) | 1.2090 (2) | 0.45413 (16) | 0.0168 (5) |
| N2 | 0.2527 (2) | 0.8608 (3) | 0.3851 (2) | 0.0309 (7) |
| H2N | 0.296 (3) | 0.785 (2) | 0.372 (3) | 0.057 (12)* |
| C1 | 0.4622 (2) | 0.6546 (3) | 0.60374 (19) | 0.0130 (6) |
| C2 | 0.5344 (2) | 0.7784 (3) | 0.6151 (2) | 0.0185 (6) |
| H2 | 0.6043 | 0.7916 | 0.5794 | 0.022* |

| | | | | |
|---|---|---|---|---|
| C3 | 0.5038 (3) | 0.8816 (3) | 0.6784 (2) | 0.0232 (7) |
| H3 | 0.5529 | 0.9654 | 0.6862 | 0.028* |
| C4 | 0.4025 (3) | 0.8631 (3) | 0.7301 (2) | 0.0248 (7) |
| H4 | 0.3807 | 0.9354 | 0.7722 | 0.030* |
| C5 | 0.3325 (3) | 0.7406 (3) | 0.7212 (2) | 0.0219 (7) |
| H5 | 0.2639 | 0.7277 | 0.7582 | 0.026* |
| C6 | 0.3615 (2) | 0.6360 (3) | 0.65865 (19) | 0.0172 (6) |
| H6 | 0.3131 | 0.5515 | 0.6530 | 0.021* |
| C7 | −0.0284 (3) | 1.2845 (3) | 0.3844 (2) | 0.0260 (7) |
| H7A | 0.0236 | 1.3368 | 0.3429 | 0.039* |
| H7B | −0.0816 | 1.3517 | 0.4153 | 0.039* |
| H7C | −0.0804 | 1.2160 | 0.3481 | 0.039* |
| C8 | 0.0588 (3) | 1.2631 (3) | 0.5493 (2) | 0.0209 (6) |
| H8A | 0.0386 | 1.1866 | 0.5920 | 0.031* |
| H8B | −0.0001 | 1.3418 | 0.5541 | 0.031* |
| H8C | 0.1435 | 1.2972 | 0.5657 | 0.031* |
| C9 | 0.1188 (2) | 1.0979 (3) | 0.4305 (2) | 0.0141 (6) |
| C10 | 0.1155 (2) | 1.0461 (3) | 0.3381 (2) | 0.0182 (6) |
| H10 | 0.0668 | 1.0937 | 0.2900 | 0.022* |
| C11 | 0.1821 (3) | 0.9282 (3) | 0.3182 (2) | 0.0254 (7) |
| H11 | 0.1786 | 0.8932 | 0.2562 | 0.031* |
| C12 | 0.2617 (3) | 0.9093 (3) | 0.4731 (2) | 0.0280 (8) |
| H12 | 0.3141 | 0.8609 | 0.5186 | 0.034* |
| C13 | 0.1979 (2) | 1.0250 (3) | 0.4982 (2) | 0.0201 (7) |
| H13 | 0.2058 | 1.0577 | 0.5607 | 0.024* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn1 | 0.01150 (12) | 0.01286 (14) | 0.01055 (14) | 0.00047 (10) | 0.00166 (10) | 0.00068 (11) |
| Br1 | 0.01193 (13) | 0.01943 (15) | 0.01628 (15) | 0.00059 (10) | 0.00046 (10) | 0.00031 (12) |
| Br2 | 0.01807 (13) | 0.01534 (14) | 0.01326 (14) | −0.00163 (11) | 0.00254 (10) | 0.00332 (13) |
| N1 | 0.0165 (11) | 0.0180 (12) | 0.0155 (13) | 0.0016 (10) | −0.0018 (9) | 0.0031 (11) |
| N2 | 0.0372 (15) | 0.0258 (16) | 0.0305 (17) | 0.0166 (13) | 0.0093 (13) | 0.0049 (14) |
| C1 | 0.0147 (13) | 0.0135 (14) | 0.0109 (15) | 0.0026 (11) | 0.0011 (11) | −0.0007 (12) |
| C2 | 0.0209 (14) | 0.0194 (15) | 0.0155 (15) | −0.0009 (12) | 0.0027 (12) | 0.0045 (13) |
| C3 | 0.0308 (16) | 0.0152 (16) | 0.0227 (17) | −0.0012 (12) | −0.0039 (13) | −0.0025 (13) |
| C4 | 0.0339 (17) | 0.0243 (17) | 0.0158 (16) | 0.0109 (14) | −0.0004 (13) | −0.0078 (14) |
| C5 | 0.0193 (14) | 0.0345 (19) | 0.0120 (15) | 0.0065 (12) | 0.0012 (12) | −0.0018 (14) |
| C6 | 0.0139 (13) | 0.0235 (16) | 0.0138 (15) | 0.0001 (11) | −0.0015 (11) | −0.0003 (13) |
| C7 | 0.0217 (15) | 0.0307 (18) | 0.0244 (17) | 0.0081 (13) | −0.0072 (13) | −0.0020 (15) |
| C8 | 0.0233 (14) | 0.0217 (16) | 0.0178 (16) | −0.0016 (12) | 0.0018 (12) | −0.0022 (13) |
| C9 | 0.0132 (12) | 0.0147 (14) | 0.0147 (15) | −0.0048 (10) | 0.0016 (11) | 0.0038 (12) |
| C10 | 0.0184 (14) | 0.0163 (15) | 0.0197 (16) | 0.0006 (11) | 0.0000 (12) | 0.0056 (13) |
| C11 | 0.0345 (17) | 0.0237 (17) | 0.0188 (17) | 0.0052 (14) | 0.0073 (14) | 0.0021 (15) |
| C12 | 0.0271 (16) | 0.0303 (19) | 0.0261 (19) | 0.0057 (14) | −0.0014 (14) | 0.0102 (16) |
| C13 | 0.0192 (13) | 0.0234 (17) | 0.0175 (16) | −0.0012 (12) | 0.0010 (12) | 0.0045 (13) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Sn1—C1 | 2.143 (3) | C4—H4 | 0.9500 |
| Sn1—C1$^{i}$ | 2.143 (3) | C5—C6 | 1.384 (4) |
| Sn1—Br1 | 2.7395 (2) | C5—H5 | 0.9500 |
| Sn1—Br1$^{i}$ | 2.7395 (2) | C6—H6 | 0.9500 |
| Sn1—Br2 | 2.7470 (3) | C7—H7A | 0.9800 |
| Sn1—Br2$^{i}$ | 2.7470 (3) | C7—H7B | 0.9800 |
| N1—C9 | 1.334 (3) | C7—H7C | 0.9800 |
| N1—C7 | 1.454 (3) | C8—H8A | 0.9800 |
| N1—C8 | 1.459 (4) | C8—H8B | 0.9800 |
| N2—C12 | 1.344 (4) | C8—H8C | 0.9800 |
| N2—C11 | 1.342 (4) | C9—C10 | 1.415 (4) |
| N2—H2N | 0.879 (10) | C9—C13 | 1.424 (4) |
| C1—C6 | 1.399 (4) | C10—C11 | 1.361 (4) |
| C1—C2 | 1.402 (4) | C10—H10 | 0.9500 |
| C2—C3 | 1.387 (4) | C11—H11 | 0.9500 |
| C2—H2 | 0.9500 | C12—C13 | 1.349 (4) |
| C3—C4 | 1.376 (4) | C12—H12 | 0.9500 |
| C3—H3 | 0.9500 | C13—H13 | 0.9500 |
| C4—C5 | 1.376 (4) | | |
| C1—Sn1—C1$^{i}$ | 180.0 | C6—C5—C4 | 120.3 (3) |
| C1—Sn1—Br1 | 90.53 (7) | C6—C5—H5 | 119.8 |
| C1—Sn1—Br1$^{i}$ | 89.47 (7) | C4—C5—H5 | 119.8 |
| C1—Sn1—Br2 | 89.64 (7) | C5—C6—C1 | 120.1 (3) |
| C1—Sn1—Br2$^{i}$ | 90.36 (7) | C5—C6—H6 | 120.0 |
| C1$^{i}$—Sn1—Br1 | 89.47 (7) | C1—C6—H6 | 120.0 |
| C1$^{i}$—Sn1—Br1$^{i}$ | 90.53 (7) | N1—C7—H7A | 109.5 |
| C1$^{i}$—Sn1—Br2 | 90.36 (7) | N1—C7—H7B | 109.5 |
| C1$^{i}$—Sn1—Br2$^{i}$ | 89.64 (7) | H7A—C7—H7B | 109.5 |
| Br1—Sn1—Br1$^{i}$ | 180.0 | N1—C7—H7C | 109.5 |
| Br1—Sn1—Br2 | 88.981 (8) | H7A—C7—H7C | 109.5 |
| Br1—Sn1—Br2$^{i}$ | 91.019 (8) | H7B—C7—H7C | 109.5 |
| Br1$^{i}$—Sn1—Br2$^{i}$ | 88.981 (8) | N1—C8—H8A | 109.5 |
| Br1$^{i}$—Sn1—Br2 | 91.019 (8) | N1—C8—H8B | 109.5 |
| Br2—Sn1—Br2$^{i}$ | 180.0 | H8A—C8—H8B | 109.5 |
| C9—N1—C7 | 120.7 (2) | N1—C8—H8C | 109.5 |
| C9—N1—C8 | 121.0 (2) | H8A—C8—H8C | 109.5 |
| C7—N1—C8 | 118.2 (2) | H8B—C8—H8C | 109.5 |
| C12—N2—C11 | 121.0 (3) | N1—C9—C10 | 122.1 (2) |
| C12—N2—H2N | 118 (3) | N1—C9—C13 | 121.0 (3) |
| C11—N2—H2N | 120 (3) | C10—C9—C13 | 116.9 (2) |
| C6—C1—C2 | 118.9 (3) | C11—C10—C9 | 120.0 (3) |
| C6—C1—Sn1 | 120.3 (2) | C11—C10—H10 | 120.0 |
| C2—C1—Sn1 | 120.71 (19) | C9—C10—H10 | 120.0 |

| | | | |
|---|---|---|---|
| C3—C2—C1 | 120.0 (3) | N2—C11—C10 | 120.8 (3) |
| C3—C2—H2 | 120.0 | N2—C11—H11 | 119.6 |
| C1—C2—H2 | 120.0 | C10—C11—H11 | 119.6 |
| C4—C3—C2 | 120.2 (3) | N2—C12—C13 | 121.5 (3) |
| C4—C3—H3 | 119.9 | N2—C12—H12 | 119.3 |
| C2—C3—H3 | 119.9 | C13—C12—H12 | 119.3 |
| C3—C4—C5 | 120.4 (3) | C12—C13—C9 | 119.7 (3) |
| C3—C4—H4 | 119.8 | C12—C13—H13 | 120.1 |
| C5—C4—H4 | 119.8 | C9—C13—H13 | 120.1 |
| Br1$^{i}$—Sn1—C1—C6 | −130.5 (2) | C2—C1—C6—C5 | 1.7 (4) |
| Br1—Sn1—C1—C6 | 49.5 (2) | Sn1—C1—C6—C5 | −175.7 (2) |
| Br2$^{i}$—Sn1—C1—C6 | −41.5 (2) | C7—N1—C9—C10 | −1.6 (4) |
| Br2—Sn1—C1—C6 | 138.5 (2) | C8—N1—C9—C10 | −178.0 (2) |
| Br1$^{i}$—Sn1—C1—C2 | 52.2 (2) | C7—N1—C9—C13 | 178.9 (2) |
| Br1—Sn1—C1—C2 | −127.8 (2) | C8—N1—C9—C13 | 2.6 (4) |
| Br2$^{i}$—Sn1—C1—C2 | 141.2 (2) | N1—C9—C10—C11 | −176.8 (3) |
| Br2—Sn1—C1—C2 | −38.8 (2) | C13—C9—C10—C11 | 2.6 (4) |
| C6—C1—C2—C3 | −1.5 (4) | C12—N2—C11—C10 | −1.4 (5) |
| Sn1—C1—C2—C3 | 175.9 (2) | C9—C10—C11—N2 | −0.9 (4) |
| C1—C2—C3—C4 | −0.2 (4) | C11—N2—C12—C13 | 1.7 (5) |
| C2—C3—C4—C5 | 1.7 (4) | N2—C12—C13—C9 | 0.2 (5) |
| C3—C4—C5—C6 | −1.5 (4) | N1—C9—C13—C12 | 177.1 (3) |
| C4—C5—C6—C1 | −0.2 (4) | C10—C9—C13—C12 | −2.3 (4) |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2N···Br1 | 0.88 (1) | 2.79 (3) | 3.385 (3) | 126 (3) |
| N2—H2N···Br2 | 0.88 (1) | 2.81 (3) | 3.485 (3) | 135 (3) |

**Fig. 1**