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## Structure Reports

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

Kong Mun Lo and Seik Weng Ng\*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

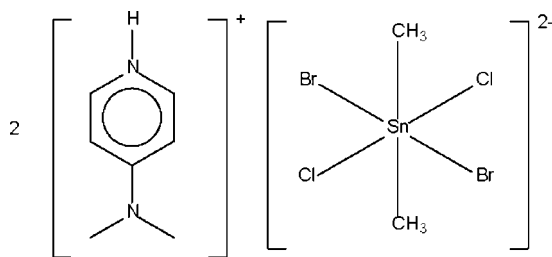
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å; disorder in main residue;  $R$  factor = 0.046;  $wR$  factor = 0.124; data-to-parameter ratio = 22.6.

The tin(IV) atom in the salt,  $(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_2(\text{CH}_3)_2\text{Cl}_2]$ , lies on a center of inversion in a tetragonally compressed octahedron; the bromine atoms are disordered with the chlorine atoms, so that they appear to share the same site. The crystal structure is stabilized by  $\text{N}-\text{H}\cdots\text{Br}$  hydrogen bonds.

## Related literature

For the structure of bis(4-dimethylaminopyridinium) tetrabromidodiphenylstannate(IV), see: Yap *et al.* (2008).



## Experimental

### Crystal data

 $(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_2(\text{CH}_3)_2\text{Cl}_2]$  $M_r = 625.83$ Triclinic,  $P\bar{1}$  $a = 7.3573$  (3) Å $b = 8.7717$  (3) Å $c = 9.6644$  (4) Å $\alpha = 97.183$  (3)° $\beta = 107.990$  (3)° $\gamma = 90.052$  (2)° $V = 588.04$  (4) Å<sup>3</sup> $Z = 1$ Mo  $K\alpha$  radiation $\mu = 4.72$  mm<sup>-1</sup> $T = 100$  (2) K

0.25 × 0.20 × 0.15 mm

### Data collection

Bruker SMART APEX  
diffractometerAbsorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996) $T_{\min} = 0.385$ ,  $T_{\max} = 0.538$ 

(expected range = 0.353–0.493)

4965 measured reflections

2756 independent reflections

1656 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.045$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$  $wR(F^2) = 0.123$  $S = 1.04$ 

2756 reflections

122 parameters

4 restraints

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.82$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -1.14$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Sn1—C1	2.225 (5)	Sn1—X2	2.6926 (8)
Sn1—X1	2.690 (1)		

X is a disordered mixture of Cl and Br.

**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 $\cdots$ X1	0.88	2.60	3.316 (5)	139
N1—H1 $\cdots$ X2 <sup>i</sup>	0.88	2.81	3.458 (6)	132

X is a disordered mixture of Cl and Br. Symmetry code: (i)  $-x + 1, -y + 1, -z + 1$ .

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: *pubCIF* (Westrip, 2008).

We thank the University of Malaya for funding this study (SF022155/2007 A) and also for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2709).

## References

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## supporting information

*Acta Cryst.* (2008). E64, m800 [doi:10.1107/S1600536808013561]

**Bis[4-(dimethylamino)pyridinium] dibromidodichloridodimethylstannate(IV)****Kong Mun Lo and Seik Weng Ng****S1. Comment**

Bis(4-methylaminopyridinium) tetrabromidodiphenylstannate is produced from the cleavage of the mixed alkyl/triaryl-stannate, cyclopentyltriphenyltin, by 4-dimethylaminopyridine hydrobromide perbromide (Yap *et al.*, 2008). In principle, the salt can be synthesized from the reaction of 4-dimethylaminopyridine hydrobromide perbromide and diphenyltin dibromide. The possibility is borne out by reacting the organic reagent with dimethyltin dichloride to yield the title salt (Scheme I, Fig. 1). The Sn<sup>IV</sup> atom of the stannate lies on a center-of-inversion in tetragonally compressed octahedron; the two independent bromine atom share the same site as the two independent chlorine atoms.

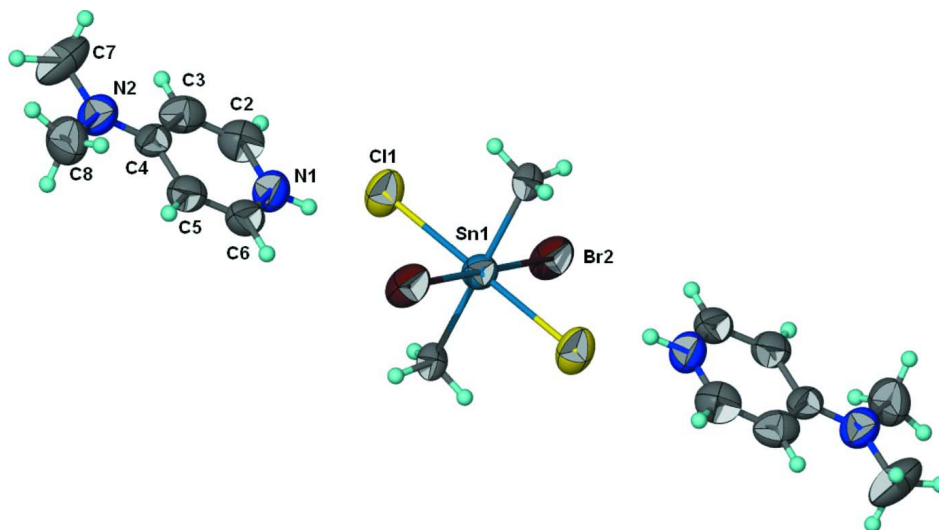
**S2. Experimental**

Dimethyltin dichloride (2.20 g, 1 mmol) and 4-dimethylaminopyridine hydrobromide perbromide (3.62 g, 1 mmol) were heated in ethanol in an attempt to synthesize the bromodichloridodimethylstannate salt. Colorless crystals separated from it after a few days.

**S3. 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  $U(\text{H})$  set to 1.2 to 1.5  $U_{\text{eq}}(\text{C})$ . The ammonium H atom was similarly treated (N—H 0.88 Å;  $U(\text{H}) = 1.2 U_{\text{eq}}(\text{N})$ ).

The chlorine atoms are disordered with respect to the bromine atoms, so that the halogen site is occupied by both a chlorine and a bromine. Constraints were applied so that at each site, the atoms had the same coordinates and the same anisotropic displacement parameters. The occupancies refined to 0.4551 (15) for the Br1/Cl2 pair, and to 0.5449 (15) for the Br2/Cl1 pair. The final difference Fourier map had a large peak at 1 Å from Sn1.

**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) plot of  $[\text{C}_7\text{H}_{11}\text{N}_2][\text{SnBr}_2\text{Cl}_2(\text{CH}_3)_2]$  at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The disorder is not shown.

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

#### Crystal data

$(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_2(\text{CH}_3)_2\text{Cl}_2]$

$M_r = 625.83$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.3573$  (3) Å

$b = 8.7717$  (3) Å

$c = 9.6644$  (4) Å

$\alpha = 97.183$  (3)°

$\beta = 107.990$  (3)°

$\gamma = 90.052$  (2)°

$V = 588.04$  (4) Å<sup>3</sup>

$Z = 1$

$F(000) = 306$

$D_x = 1.767$  Mg m<sup>-3</sup>

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

Cell parameters from 1302 reflections

$\theta = 2.3$ – $23.1$ °

$\mu = 4.72$  mm<sup>-1</sup>

$T = 100$  K

Prism, colorless

$0.25 \times 0.20 \times 0.15$  mm

#### Data collection

Bruker SMART APEX  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.385$ ,  $T_{\max} = 0.538$

4965 measured reflections

2756 independent reflections

1656 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.5$ °,  $\theta_{\min} = 2.3$ °

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 11$

$l = -10 \rightarrow 12$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.123$

$S = 1.04$

2756 reflections

122 parameters

4 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

$$w = 1/[\sigma^2(F_o^2) + (0.051P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$$\Delta\rho_{\max} = 0.82 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.14 \text{ e } \text{\AA}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Sn1	0.5000	0.5000	0.5000	0.0414 (2)	
Br1	0.50474 (16)	0.50403 (11)	0.77987 (11)	0.0610 (3)	0.4551 (15)
Br2	0.36593 (14)	0.78543 (10)	0.49452 (10)	0.0588 (3)	0.5449 (15)
Cl1	0.50474 (16)	0.50403 (11)	0.77987 (11)	0.0610 (3)	0.5449 (15)
Cl2	0.36593 (14)	0.78543 (10)	0.49452 (10)	0.0588 (3)	0.4551 (15)
N1	0.6576 (7)	0.1602 (6)	0.8588 (6)	0.0552 (14)	
H1	0.6205	0.2248	0.7938	0.066*	
N2	0.8414 (7)	-0.1391 (6)	1.1635 (6)	0.0557 (14)	
C1	0.2014 (7)	0.4016 (6)	0.4157 (6)	0.0344 (12)	
H1A	0.2023	0.2904	0.4192	0.052*	
H1B	0.1432	0.4214	0.3142	0.052*	
H1C	0.1271	0.4495	0.4767	0.052*	
C2	0.6816 (9)	0.2092 (8)	1.0006 (8)	0.0581 (18)	
H2	0.6571	0.3128	1.0290	0.070*	
C3	0.7413 (9)	0.1116 (7)	1.1061 (7)	0.0545 (16)	
H3	0.7570	0.1480	1.2060	0.065*	
C4	0.7793 (8)	-0.0433 (7)	1.0650 (7)	0.0443 (15)	
C5	0.7486 (8)	-0.0875 (7)	0.9140 (7)	0.0488 (15)	
H5	0.7698	-0.1904	0.8805	0.059*	
C6	0.6890 (9)	0.0144 (8)	0.8144 (7)	0.0536 (16)	
H6	0.6698	-0.0183	0.7132	0.064*	
C7	0.8687 (11)	-0.0920 (10)	1.3200 (8)	0.084 (3)	
H7A	0.9886	-0.0305	1.3639	0.127*	
H7B	0.8740	-0.1836	1.3695	0.127*	
H7C	0.7616	-0.0305	1.3310	0.127*	
C8	0.8809 (10)	-0.2972 (8)	1.1236 (9)	0.071 (2)	
H8A	0.7602	-0.3560	1.0736	0.106*	
H8B	0.9498	-0.3417	1.2124	0.106*	
H8C	0.9594	-0.3008	1.0579	0.106*	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.0437 (4)	0.0408 (3)	0.0388 (4)	0.0028 (3)	0.0122 (3)	0.0040 (3)
Br1	0.0938 (9)	0.0501 (6)	0.0460 (6)	0.0082 (5)	0.0319 (6)	0.0062 (5)
Br2	0.0809 (7)	0.0458 (5)	0.0461 (6)	0.0187 (5)	0.0144 (5)	0.0062 (4)
Cl1	0.0938 (9)	0.0501 (6)	0.0460 (6)	0.0082 (5)	0.0319 (6)	0.0062 (5)
Cl2	0.0809 (7)	0.0458 (5)	0.0461 (6)	0.0187 (5)	0.0144 (5)	0.0062 (4)
N1	0.050 (3)	0.064 (4)	0.052 (4)	0.001 (3)	0.012 (3)	0.020 (3)
N2	0.048 (3)	0.070 (4)	0.050 (3)	0.004 (3)	0.012 (3)	0.017 (3)
C1	0.032 (3)	0.039 (3)	0.040 (3)	0.009 (2)	0.019 (3)	0.012 (3)

C2	0.050 (4)	0.058 (4)	0.064 (5)	0.007 (3)	0.016 (4)	0.004 (4)
C3	0.048 (4)	0.065 (4)	0.048 (4)	0.001 (3)	0.016 (3)	-0.006 (3)
C4	0.031 (3)	0.063 (4)	0.037 (3)	-0.005 (3)	0.008 (3)	0.009 (3)
C5	0.047 (4)	0.050 (3)	0.047 (4)	-0.003 (3)	0.013 (3)	0.002 (3)
C6	0.054 (4)	0.064 (4)	0.042 (4)	-0.013 (3)	0.016 (3)	0.001 (3)
C7	0.075 (5)	0.136 (7)	0.043 (4)	0.025 (5)	0.013 (4)	0.028 (5)
C8	0.071 (5)	0.063 (5)	0.079 (6)	0.002 (4)	0.016 (4)	0.026 (4)

*Geometric parameters (Å, °)*

Sn1—C1 <sup>i</sup>	2.225 (5)	C1—H1C	0.9800
Sn1—C1	2.225 (5)	C2—C3	1.381 (8)
Sn1—Br1	2.690 (1)	C2—H2	0.9500
Sn1—Cl1 <sup>i</sup>	2.690 (1)	C3—C4	1.420 (9)
Sn1—Br1 <sup>i</sup>	2.690 (1)	C3—H3	0.9500
Sn1—Br2 <sup>i</sup>	2.6926 (8)	C4—C5	1.409 (9)
Sn1—Br2	2.6926 (8)	C5—C6	1.369 (8)
Sn1—Cl2 <sup>i</sup>	2.6926 (8)	C5—H5	0.9500
N1—C2	1.341 (9)	C6—H6	0.9500
N1—C6	1.341 (9)	C7—H7A	0.9800
N1—H1	0.8800	C7—H7B	0.9800
N2—C4	1.324 (7)	C7—H7C	0.9800
N2—C8	1.446 (9)	C8—H8A	0.9800
N2—C7	1.467 (9)	C8—H8B	0.9800
C1—H1A	0.9800	C8—H8C	0.9800
C1—H1B	0.9800		
C1 <sup>i</sup> —Sn1—C1	180.0	Sn1—C1—H1A	109.5
C1 <sup>i</sup> —Sn1—Br1	88.38 (14)	Sn1—C1—H1B	109.5
C1—Sn1—Br1	91.62 (14)	H1A—C1—H1B	109.5
C1 <sup>i</sup> —Sn1—Cl1 <sup>i</sup>	91.62 (14)	Sn1—C1—H1C	109.5
C1—Sn1—Cl1 <sup>i</sup>	88.38 (14)	H1A—C1—H1C	109.5
Br1—Sn1—Cl1 <sup>i</sup>	180.0	H1B—C1—H1C	109.5
C1 <sup>i</sup> —Sn1—Br1 <sup>i</sup>	91.62 (14)	N1—C2—C3	121.1 (6)
C1—Sn1—Br1 <sup>i</sup>	88.38 (14)	N1—C2—H2	119.5
Br1—Sn1—Br1 <sup>i</sup>	180.0	C3—C2—H2	119.5
Cl1 <sup>i</sup> —Sn1—Br1 <sup>i</sup>	0.0	C2—C3—C4	120.0 (6)
C1 <sup>i</sup> —Sn1—Br2 <sup>i</sup>	89.83 (13)	C2—C3—H3	120.0
C1—Sn1—Br2 <sup>i</sup>	90.17 (13)	C4—C3—H3	120.0
Br1—Sn1—Br2 <sup>i</sup>	89.12 (3)	N2—C4—C5	122.5 (6)
Cl1 <sup>i</sup> —Sn1—Br2 <sup>i</sup>	90.88 (3)	N2—C4—C3	121.6 (6)
Br1 <sup>i</sup> —Sn1—Br2 <sup>i</sup>	90.88 (3)	C5—C4—C3	115.9 (6)
C1 <sup>i</sup> —Sn1—Br2	90.17 (13)	C6—C5—C4	121.5 (6)
C1—Sn1—Br2	89.83 (13)	C6—C5—H5	119.2
Br1—Sn1—Br2	90.88 (3)	C4—C5—H5	119.2
Cl1 <sup>i</sup> —Sn1—Br2	89.12 (3)	N1—C6—C5	120.4 (6)
Br1 <sup>i</sup> —Sn1—Br2	89.12 (3)	N1—C6—H6	119.8
Br2 <sup>i</sup> —Sn1—Br2	180.0	C5—C6—H6	119.8

C1 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	89.83 (13)	N2—C7—H7A	109.5
C1—Sn1—Cl2 <sup>i</sup>	90.17 (13)	N2—C7—H7B	109.5
Br1—Sn1—Cl2 <sup>i</sup>	89.12 (3)	H7A—C7—H7B	109.5
Cl1 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	90.88 (3)	N2—C7—H7C	109.5
Br1 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	90.88 (3)	H7A—C7—H7C	109.5
Br2 <sup>i</sup> —Sn1—Cl2 <sup>i</sup>	0.000 (13)	H7B—C7—H7C	109.5
Br2—Sn1—Cl2 <sup>i</sup>	180.0	N2—C8—H8A	109.5
C2—N1—C6	121.1 (6)	N2—C8—H8B	109.5
C2—N1—H1	119.4	H8A—C8—H8B	109.5
C6—N1—H1	119.4	N2—C8—H8C	109.5
C4—N2—C8	122.3 (6)	H8A—C8—H8C	109.5
C4—N2—C7	121.8 (6)	H8B—C8—H8C	109.5
C8—N2—C7	115.9 (6)		
C6—N1—C2—C3	0.6 (9)	C2—C3—C4—N2	178.3 (5)
N1—C2—C3—C4	0.3 (10)	C2—C3—C4—C5	-1.1 (8)
C8—N2—C4—C5	-0.6 (9)	N2—C4—C5—C6	-178.3 (6)
C7—N2—C4—C5	-178.7 (6)	C3—C4—C5—C6	1.1 (9)
C8—N2—C4—C3	-180.0 (5)	C2—N1—C6—C5	-0.7 (9)
C7—N2—C4—C3	1.9 (9)	C4—C5—C6—N1	-0.2 (9)

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

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ Br1	0.88	2.60	3.316 (5)	139
N1—H1 $\cdots$ Br2 <sup>i</sup>	0.88	2.81	3.458 (6)	132

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