

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis[4-(dimethylamino)pyridinium]hexakis[bromido/chlorido(0.78/0.22)]stannate(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

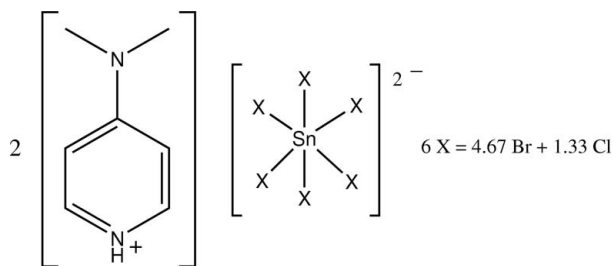
Received 19 May 2009; accepted 25 May 2009

 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.023; wR factor = 0.060; data-to-parameter ratio = 20.6.

The Sn atom in the title salt, $(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_{4.67}\text{Cl}_{1.33}]$, lies on a center of symmetry within an octahedron of disordered halogen atoms. The three independent halogen atoms are each a mixture of bromine and chlorine atoms [with site occupancies for bromine of 0.614 (1), 0.831 (1) and 0.888 (1)]. An $\text{N}-\text{H}\cdots$ hydrogen bond is present.

Related literature

For the isostructural tribromidotrichloridostannate, see: Lo & Ng (2008); for the isostructural pentabromidochloridostannate, see: Jang *et al.* (2009).



Experimental

Crystal data

 $(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_{4.67}\text{Cl}_{1.33}]$
 $M_r = 785.15$

 Monoclinic, $P2_1/c$
 $a = 8.4530$ (2) Å

 $b = 11.9036$ (2) Å

 $c = 11.9093$ (2) Å

 $\beta = 107.109$ (1)°

 $V = 1145.30$ (4) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 9.42$ mm⁻¹
 $T = 100$ K

 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART APEX

diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.504$, $T_{\max} = 0.746$

(expected range = 0.103–0.152)

10319 measured reflections

2622 independent reflections

 2240 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.060$
 $S = 0.99$

2622 reflections

127 parameters

6 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.80$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.87$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{Br1}$	0.88 (1)	2.484 (18)	3.334 (3)	162 (4)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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, 2009).

We thank the University of Malaya (RG020/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2458).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Bruker (2007). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Jang, Y., Lo, K. M. & Ng, S. W. (2009). *Acta Cryst.* **E65**, m645.
 Lo, K. M. & Ng, S. W. (2008). *Acta Cryst.* **E64**, m834.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, m715 [doi:10.1107/S1600536809019734]

Bis[4-(dimethylamino)pyridinium] hexakis[bromido/chlorido(0.78/0.22)]stannate(IV)

K. M. Lo and S. W. Ng

Experimental

Dibenzyltin dichloride (0.37 g, 1 mmol) and 4-dimethylaminopyridine hydrobromide perbromide (0.73 g, 2 mmol) were heated in chloroform for 1 hour. Colorless crystals separated from the cool solution after a day. The benzyl groups on tin has been cleaved in the reaction. In the previous study, a heating time of 3 hours gave the pentabromidochloridostannate (Jang *et al.*, 2009).

Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95–0.98, N–H 0.88 Å) and were treated as riding on their parent atoms, with $U(H)$ set to 1.2–1.5 times $U_{eq}(C,N)$.

The three halogen atoms in the stannate are disordered. The sum of the occupancies of the three bromide atoms refined to nearly 2.33Br and 0.67Cl atoms; the total occupancy of the disordered bromide atoms was then fixed as exactly 2.333. The occupancy of the disordered chloride atoms was similarly set to be 0.667. The anisotropic displacement parameters of each pair of Br/Cl atoms were restrained to be identical.

Figures

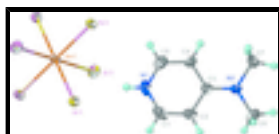


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $2[C_7H_{11}N_2]^+ [SnBr_{4.67}Cl_{1.33}]^{2-}$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The bromine atoms are disordered with respect to the chlorine atoms.

Bis[4-(dimethylamino)pyridinium] hexakis[bromido/chlorido(0.78/0.22)]stannate(IV)

Crystal data

$(C_7H_{11}N_2)_2[SnBr_{4.67}Cl_{1.33}]$

$M_r = 785.15$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.4530\ (2)\ \text{\AA}$

$b = 11.9036\ (2)\ \text{\AA}$

$c = 11.9093\ (2)\ \text{\AA}$

$\beta = 107.109\ (1)^\circ$

$V = 1145.30\ (4)\ \text{\AA}^3$

$Z = 2$

$F_{000} = 740$

$D_x = 2.277\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 4263 reflections

$\theta = 2.5\text{--}28.3^\circ$

$\mu = 9.42\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Irregular block, colorless

$0.30 \times 0.25 \times 0.20\ \text{mm}$

supplementary materials

Data collection

Bruker SMART APEX diffractometer	2622 independent reflections
Radiation source: fine-focus sealed tube	2240 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.033$
$T = 100$ K	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 2.5^\circ$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.504$, $T_{\text{max}} = 0.746$	$k = -15 \rightarrow 15$
10319 measured reflections	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.023$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.6005P]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
2622 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
127 parameters	$\Delta\rho_{\text{max}} = 0.80 \text{ e } \text{\AA}^{-3}$
6 restraints	$\Delta\rho_{\text{min}} = -0.87 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Sn1	0.5000	0.5000	0.5000	0.01384 (8)	
Br1	0.50914 (5)	0.63592 (3)	0.66901 (3)	0.02247 (12)	0.6143 (14)
Br2	0.58481 (5)	0.33875 (3)	0.64866 (3)	0.02405 (12)	0.8309 (9)
Br3	0.80683 (4)	0.53911 (3)	0.52227 (3)	0.02760 (12)	0.8878 (10)
Cl1	0.50914 (5)	0.63592 (3)	0.66901 (3)	0.02247 (12)	0.3858 (14)
Cl2	0.58481 (5)	0.33875 (3)	0.64866 (3)	0.02405 (12)	0.1122 (10)
Cl3	0.80683 (4)	0.53911 (3)	0.52227 (3)	0.02760 (12)	0.1691 (9)
N1	0.6521 (4)	0.8743 (2)	0.5886 (3)	0.0309 (7)	
H1	0.598 (5)	0.812 (2)	0.593 (4)	0.061 (14)*	
N2	0.9135 (3)	1.1561 (2)	0.5550 (2)	0.0231 (6)	
C1	0.7281 (4)	0.9350 (3)	0.6844 (3)	0.0304 (8)	
H1A	0.7212	0.9116	0.7590	0.036*	
C2	0.8143 (4)	1.0288 (3)	0.6765 (3)	0.0259 (7)	
H2	0.8683	1.0696	0.7457	0.031*	
C3	0.8251 (4)	1.0670 (3)	0.5661 (3)	0.0190 (6)	

C4	0.7363 (4)	1.0019 (3)	0.4663 (3)	0.0228 (7)
H4	0.7345	1.0249	0.3896	0.027*
C5	0.6553 (4)	0.9080 (3)	0.4810 (3)	0.0297 (8)
H5	0.5994	0.8645	0.4142	0.036*
C6	1.0103 (4)	1.2201 (3)	0.6573 (3)	0.0349 (8)
H6A	0.9356	1.2652	0.6883	0.052*
H6B	1.0876	1.2697	0.6340	0.052*
H6C	1.0725	1.1680	0.7182	0.052*
C7	0.9124 (4)	1.1989 (3)	0.4397 (3)	0.0293 (7)
H7A	0.9577	1.1418	0.3985	0.044*
H7B	0.9802	1.2670	0.4496	0.044*
H7C	0.7985	1.2166	0.3937	0.044*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01350 (14)	0.01560 (14)	0.01208 (14)	-0.00189 (10)	0.00326 (11)	-0.00020 (11)
Br1	0.0329 (2)	0.0187 (2)	0.0178 (2)	-0.00322 (16)	0.01055 (17)	-0.00423 (15)
Br2	0.0329 (2)	0.01902 (19)	0.01671 (19)	-0.00065 (14)	0.00180 (15)	0.00479 (13)
Br3	0.01394 (18)	0.0409 (2)	0.0279 (2)	-0.00705 (13)	0.00609 (14)	-0.00114 (15)
Cl1	0.0329 (2)	0.0187 (2)	0.0178 (2)	-0.00322 (16)	0.01055 (17)	-0.00423 (15)
Cl2	0.0329 (2)	0.01902 (19)	0.01671 (19)	-0.00065 (14)	0.00180 (15)	0.00479 (13)
Cl3	0.01394 (18)	0.0409 (2)	0.0279 (2)	-0.00705 (13)	0.00609 (14)	-0.00114 (15)
N1	0.0277 (16)	0.0246 (15)	0.0427 (19)	0.0002 (12)	0.0141 (14)	0.0088 (14)
N2	0.0235 (14)	0.0244 (14)	0.0195 (14)	-0.0029 (11)	0.0032 (11)	-0.0017 (11)
C1	0.0298 (19)	0.037 (2)	0.0273 (18)	0.0121 (15)	0.0138 (15)	0.0114 (16)
C2	0.0256 (17)	0.0335 (18)	0.0190 (17)	0.0045 (14)	0.0071 (14)	0.0010 (13)
C3	0.0157 (14)	0.0217 (15)	0.0185 (15)	0.0046 (11)	0.0036 (12)	0.0009 (12)
C4	0.0214 (15)	0.0252 (16)	0.0198 (16)	-0.0003 (13)	0.0030 (13)	-0.0026 (13)
C5	0.0238 (17)	0.0282 (18)	0.033 (2)	-0.0001 (13)	0.0021 (15)	-0.0037 (15)
C6	0.033 (2)	0.035 (2)	0.033 (2)	-0.0101 (15)	0.0035 (16)	-0.0085 (16)
C7	0.0300 (18)	0.0285 (18)	0.0270 (18)	-0.0048 (14)	0.0045 (14)	0.0082 (14)

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

Sn1—Br1	2.5658 (4)	C1—C2	1.351 (5)
Sn1—Cl1 ⁱ	2.5658 (4)	C1—H1A	0.9500
Sn1—Br1 ⁱ	2.5658 (4)	C2—C3	1.419 (4)
Sn1—Br2	2.5663 (3)	C2—H2	0.9500
Sn1—Cl2 ⁱ	2.5663 (3)	C3—C4	1.433 (4)
Sn1—Br2 ⁱ	2.5663 (3)	C4—C5	1.349 (5)
Sn1—Cl3 ⁱ	2.5709 (3)	C4—H4	0.9500
Sn1—Br3 ⁱ	2.5709 (3)	C5—H5	0.9500
Sn1—Br3	2.5709 (3)	C6—H6A	0.9800
N1—C1	1.343 (5)	C6—H6B	0.9800
N1—C5	1.351 (5)	C6—H6C	0.9800
N1—H1	0.882 (10)	C7—H7A	0.9800

supplementary materials

N2—C3	1.327 (4)	C7—H7B	0.9800
N2—C6	1.466 (4)	C7—H7C	0.9800
N2—C7	1.462 (4)		
Br1—Sn1—Cl1 ⁱ	180.0	Br3 ⁱ —Sn1—Br3	180.000 (17)
Br1—Sn1—Br1 ⁱ	180.0	C1—N1—C5	120.5 (3)
Cl1 ⁱ —Sn1—Br1 ⁱ	0.000 (14)	C1—N1—H1	122 (3)
Br1—Sn1—Br2	89.576 (13)	C5—N1—H1	118 (3)
Cl1 ⁱ —Sn1—Br2	90.424 (13)	C3—N2—C6	121.7 (3)
Br1 ⁱ —Sn1—Br2	90.424 (13)	C3—N2—C7	121.6 (3)
Br1—Sn1—Cl2 ⁱ	90.424 (13)	C6—N2—C7	116.6 (3)
Cl1 ⁱ —Sn1—Cl2 ⁱ	89.576 (12)	N1—C1—C2	121.2 (3)
Br1 ⁱ —Sn1—Cl2 ⁱ	89.576 (12)	N1—C1—H1A	119.4
Br2—Sn1—Cl2 ⁱ	180.0	C2—C1—H1A	119.4
Br1—Sn1—Br2 ⁱ	90.424 (13)	C1—C2—C3	120.8 (3)
Cl1 ⁱ —Sn1—Br2 ⁱ	89.576 (12)	C1—C2—H2	119.6
Br1 ⁱ —Sn1—Br2 ⁱ	89.576 (12)	C3—C2—H2	119.6
Br2—Sn1—Br2 ⁱ	180.0	N2—C3—C2	122.7 (3)
Cl2 ⁱ —Sn1—Br2 ⁱ	0.00 (2)	N2—C3—C4	121.5 (3)
Br1—Sn1—Cl3 ⁱ	89.529 (12)	C2—C3—C4	115.7 (3)
Cl1 ⁱ —Sn1—Cl3 ⁱ	90.471 (12)	C5—C4—C3	120.2 (3)
Br1 ⁱ —Sn1—Cl3 ⁱ	90.471 (12)	C5—C4—H4	119.9
Br2—Sn1—Cl3 ⁱ	90.248 (12)	C3—C4—H4	119.9
Cl2 ⁱ —Sn1—Cl3 ⁱ	89.752 (12)	C4—C5—N1	121.4 (3)
Br2 ⁱ —Sn1—Cl3 ⁱ	89.752 (12)	C4—C5—H5	119.3
Br1—Sn1—Br3 ⁱ	89.529 (12)	N1—C5—H5	119.3
Cl1 ⁱ —Sn1—Br3 ⁱ	90.471 (12)	N2—C6—H6A	109.5
Br1 ⁱ —Sn1—Br3 ⁱ	90.471 (12)	N2—C6—H6B	109.5
Br2—Sn1—Br3 ⁱ	90.248 (12)	H6A—C6—H6B	109.5
Cl2 ⁱ —Sn1—Br3 ⁱ	89.752 (12)	N2—C6—H6C	109.5
Br2 ⁱ —Sn1—Br3 ⁱ	89.752 (12)	H6A—C6—H6C	109.5
Cl3 ⁱ —Sn1—Br3 ⁱ	0.00 (2)	H6B—C6—H6C	109.5
Br1—Sn1—Br3	90.471 (12)	N2—C7—H7A	109.5
Cl1 ⁱ —Sn1—Br3	89.529 (12)	N2—C7—H7B	109.5
Br1 ⁱ —Sn1—Br3	89.529 (12)	H7A—C7—H7B	109.5
Br2—Sn1—Br3	89.752 (12)	N2—C7—H7C	109.5
Cl2 ⁱ —Sn1—Br3	90.248 (12)	H7A—C7—H7C	109.5
Br2 ⁱ —Sn1—Br3	90.248 (12)	H7B—C7—H7C	109.5
Cl3 ⁱ —Sn1—Br3	180.000 (17)		
C5—N1—C1—C2	-2.3 (5)	C1—C2—C3—N2	-177.4 (3)
N1—C1—C2—C3	0.9 (5)	C1—C2—C3—C4	1.5 (5)
C6—N2—C3—C2	1.6 (5)	N2—C3—C4—C5	176.1 (3)
C7—N2—C3—C2	-175.2 (3)	C2—C3—C4—C5	-2.8 (5)

C6—N2—C3—C4	-177.3 (3)	C3—C4—C5—N1	1.6 (5)
C7—N2—C3—C4	5.9 (5)	C1—N1—C5—C4	1.0 (5)

Symmetry codes: (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 (1)	2.484 (18)	3.334 (3)	162 (4)

Fig. 1

