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

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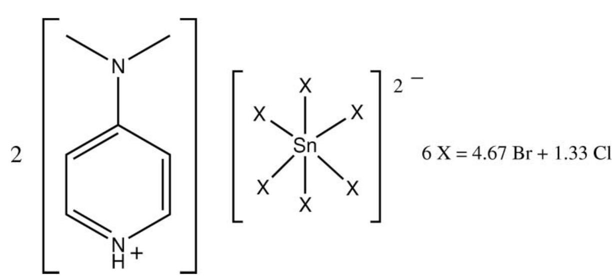
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.023; wR factor = 0.060; data-to-parameter ratio = 20.6.

The Sn atom in the title salt, $(C_7H_{11}N_2)_2[SnBr_{4.67}Cl_{1.33}]$, 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 N—H$\cdots$ hydrogen bond is present.

Related literature

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



Experimental

Crystal data

$(C_7H_{11}N_2)_2[SnBr_{4.67}Cl_{1.33}]$
M_r = 785.15
Monoclinic, $P2_1/c$
a = 8.4530 (2) Å
b = 11.9036 (2) Å
c = 11.9093 (2) Å
β = 107.109 (1)°
V = 1145.30 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 9.42 mm^{-1}
T = 100 K
0.30 × 0.25 × 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_{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
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −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$
N1—H1$\cdots$Br1	0.88 (1)	2.484 (18)	3.334 (3)	162 (4)

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, 2009).

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

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supplementary materials

Acta Cryst. (2009). E**65**, 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

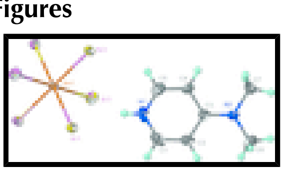

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $2[C_7H_{11}N_2]^+$ $[SnBr_{4.67}Cl_{1.33}]^{2-}$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The bromine atoms are disordered with respect to the chlorine atoms.

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

Crystal data

$(C_7H_{11}N_2)_2[SnBr_{4.67}Cl_{1.33}]$	F_{000} = 740
M_r = 785.15	D_x = 2.277 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4263 reflections
a = 8.4530 (2) Å	θ = 2.5–28.3°
b = 11.9036 (2) Å	μ = 9.42 mm^{-1}
c = 11.9093 (2) Å	T = 100 K
β = 107.109 (1)°	Irregular block, colorless
V = 1145.30 (4) Å^3	0.30 × 0.25 × 0.20 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	2622 independent reflections
Radiation source: fine-focus sealed tube	2240 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.033$
$T = 100$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.5°$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.504$, $T_{max} = 0.746$	$k = -15 \rightarrow 15$
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
$wR(F^2) = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.6005P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
2622 reflections	$\Delta\rho_{max} = 0.80$ e Å^{-3}
127 parameters	$\Delta\rho_{min} = -0.87$ e Å^{-3}
6 restraints	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}	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 (Å^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 (Å, °)

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

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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Br1	0.88 (1)	2.484 (18)	3.334 (3)	162 (4)

Fig. 1