metal-organic compounds

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

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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).
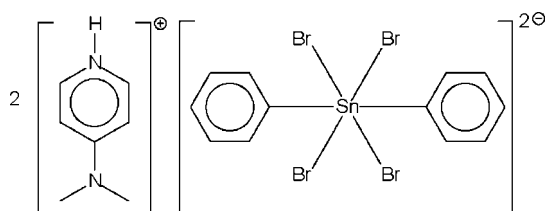
Experimental

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
$0.24 \times 0.18 \times 0.12$ 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$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2N$\cdots$Br1	0.88 (1)	2.79 (3)	3.385 (3)	126 (3)
N2—H2N$\cdots$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).

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

References

Barbour, L. J. (2001). J. Supramol. Chem. **1**, 189–191.
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Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (2008). Acta Cryst. A**64**, 112–122.
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supporting information

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

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S1. 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.

S2. 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.

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(H) set to 1.2 to 1.5U_{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.
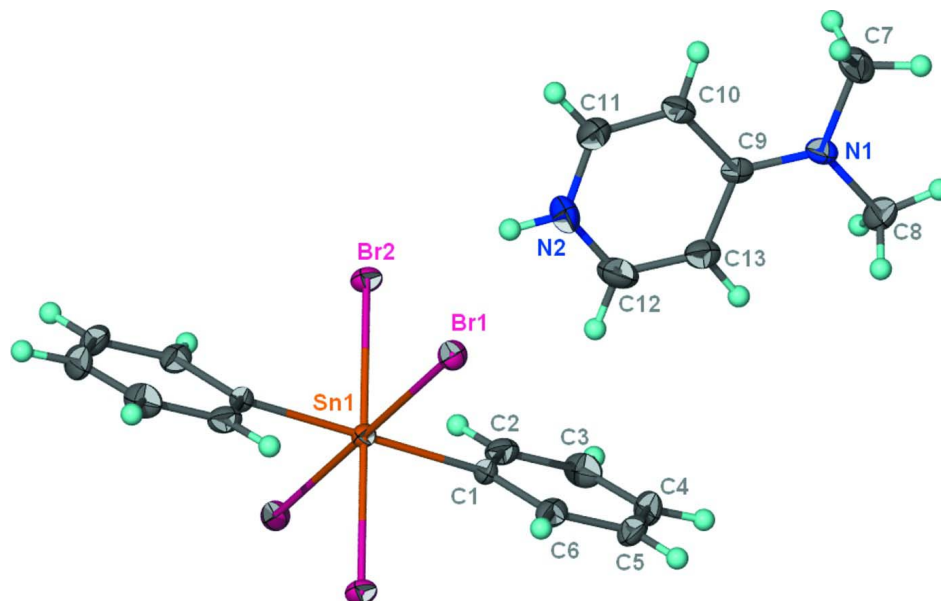
Figure 1

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, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3449 reflections
$a = 10.7803$ (2) Å	$\theta = 2.3–28.3°$
$b = 9.3847$ (2) Å	$\mu = 6.40$ mm^{-1}
$c = 14.4068$ (4) Å	$T = 100$ K
$\beta = 94.126$ (2)°	Block, colorless
$V = 1453.76$ (6) Å^3	$0.24 \times 0.18 \times 0.12$ mm
$Z = 2$	

Data collection

Bruker SMART APEX diffractometer	11853 measured reflections
Radiation source: fine-focus sealed tube	3334 independent reflections
Graphite monochromator	2688 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.035$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$T_{min} = 0.386$, $T_{max} = 0.514$	$h = -14 \rightarrow 14$
	$k = -12 \rightarrow 12$
	$l = -18 \rightarrow 14$

Refinement

Refinement on F^2	3334 reflections
Least-squares matrix: full	166 parameters
$R[F^2 > 2\sigma(F^2)] = 0.024$	1 restraint
$wR(F^2) = 0.051$	Primary atom site location: structure-invariant
$S = 0.99$	direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0228P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

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, º)

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

C1ⁱ—Sn1—Br2ⁱ	89.64 (7)	H7A—C7—H7B	109.5
Br1—Sn1—Br1ⁱ	180.0	N1—C7—H7C	109.5
Br1—Sn1—Br2	88.981 (8)	H7A—C7—H7C	109.5
Br1—Sn1—Br2ⁱ	91.019 (8)	H7B—C7—H7C	109.5
Br1ⁱ—Sn1—Br2ⁱ	88.981 (8)	N1—C8—H8A	109.5
Br1ⁱ—Sn1—Br2	91.019 (8)	N1—C8—H8B	109.5
Br2—Sn1—Br2ⁱ	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
Br1ⁱ—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)
Br2ⁱ—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)
Br1ⁱ—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)
Br2ⁱ—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 code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···Br1	0.88 (1)	2.79 (3)	3.385 (3)	126 (3)
N2—H2*N*···Br2	0.88 (1)	2.81 (3)	3.485 (3)	135 (3)