Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-chlorophenyl)-stannate(IV)–4-bromochlorobenzene (1/1)

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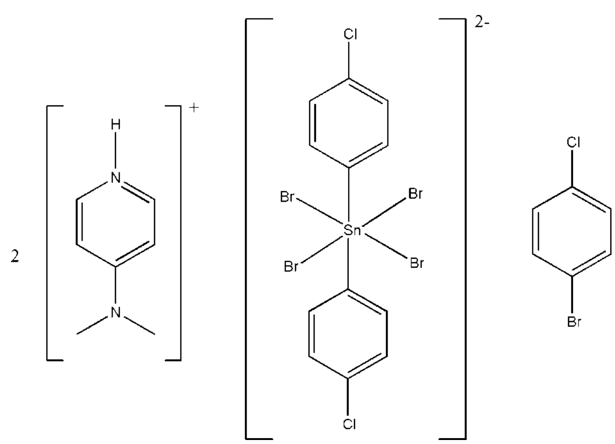
Received 8 June 2009; accepted 30 July 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; disorder in solvent or counterion; R factor = 0.023; wR factor = 0.062; data-to-parameter ratio = 20.6.

In the title compound, $(C_7H_{11}N_2)_2[SnBr_4(C_6H_4Cl)_2]\cdot C_6H_4BrCl$, the Sn^{IV} atom in the tetrabromidobis(4-chlorophenyl)stannate(IV) anion lies on a centre of inversion. The distances between the 4-(dimethylamino)pyridinium N atom and the Br atoms of the anion are 3.450 (2) and 3.452 (2) Å, suggesting weak hydrogen bonding. The 4-bromochlorobenzene solvent molecule, which is a bromination by-product from the reaction, is disordered about a twofold rotation axis with approximately equal occupancy.

Related literature

For related structures, see Lo & Ng (2009); Koon *et al.* (2009); Yap *et al.* (2008).



Experimental

Crystal data

$(C_7H_{11}N_2)_2[SnBr_4(C_6H_4Cl)_2]\cdot C_6H_4BrCl$
M_r = 1099.22
Triclinic, $P\overline{1}$
a = 8.7692 (18) Å
b = 10.128 (2) Å
c = 11.407 (2) Å
α = 111.16 (3)°
β = 93.38 (3)°
γ = 92.85 (3)°
V = 940.4 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 6.23 mm^{-1}
T = 100 K
0.45 × 0.26 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.169, T_{max} = 0.384 (expected range = 0.135–0.306)
7255 measured reflections
4265 independent reflections
3919 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.062
S = 1.05
4265 reflections
207 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −1.12 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the University of Malaya (grant Nos. PS072/2007C and PS320/2008C) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2523).

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supplementary materials

Acta Cryst. (2009). E**65**, m1039 [**doi:10.1107/S1600536809030232**]

Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-chlorophenyl)stannate(IV)-4-bromochlorobenzene (1/1)

S. M. Lee, K. M. Lo, H. Mohd Ali and W. T. Robinson

Experimental

Tetra(4-chlorophenyl)tin (0.57 g, 1 mmol) and 4-dimethylaminopyridine hydrobromide perbromide (0.40 g, 1 mmol) was dissolved in absolute ethanol (25 ml) and refluxed for six hours. The solution was filtered and colourless crystals were isolated upon cooling.

Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95 to 0.98 Å) and were treated as riding on their parent carbon atoms, with *U*(H) set to 1.2–1.5 times *U*(C,*N*). N—H was refined and placed in the calculated position of N—H 0.88 ± 0.01 Å.

Figures

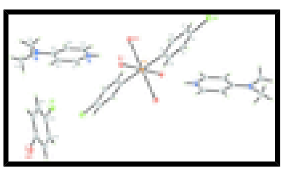


Fig. 1. The molecular structure of bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-chlorophenyl)stannate(IV) 4-bromochlorobenzene, showing 50% probability displacement ellipsoids and the atom numbering. Hydrogen atoms are drawn as spheres of arbitrary radius.

Bis[4-(dimethylamino)pyridinium] tetrabromidobis(4-chlorophenyl)stannate(IV)–4-bromochlorobenzene (1/1)

Crystal data

$(C_7H_{11}N_2)_2[SnBr_4(C_6H_4Cl_1)_2]\cdot C_6H_4BrCl$	$Z = 1$
$M_r = 1099.22$	$F_{000} = 530$
Triclinic, $P\bar{1}$	$D_x = 1.941$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.7692$ (18) Å	Cell parameters from 6036 reflections
$b = 10.128$ (2) Å	$\theta = 2.2$–30.5°
$c = 11.407$ (2) Å	$\mu = 6.23$ mm^{-1}
$\alpha = 111.16$ (3)°	$T = 100$ K
$\beta = 93.38$ (3)°	Block, colourless
$\gamma = 92.85$ (3)°	0.45 × 0.26 × 0.19 mm
$V = 940.4$ (3) Å^3	

Data collection

Bruker APEXII CCD area-detector	4265 independent reflections

diffractometer	
Radiation source: fine-focus sealed tube	3919 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 100$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 1.9°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11\to8$
$T_{min} = 0.169$, $T_{max} = 0.384$	$k = -13\to13$
7255 measured reflections	$l = -14\to13$

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-atom parameters constrained
$wR(F^2) = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.8768P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
4265 reflections	$\Delta\rho_{max} = 0.77$ e Å^{-3}
207 parameters	$\Delta\rho_{min} = -1.12$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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	0.01198 (6)	
Br1	0.43772 (3)	0.21868 (2)	0.45713 (2)	0.01709 (7)	
Br2	0.75993 (3)	0.43138 (3)	0.38027 (2)	0.01756 (7)	
Br3	0.77314 (5)	0.20503 (4)	0.97712 (4)	0.02952 (10)	0.50
Cl2	0.77314 (5)	0.20503 (4)	0.97712 (4)	0.02952 (10)	0.50
Cl1	0.13889 (10)	0.39025 (9)	−0.06015 (7)	0.03579 (19)	
N1	−0.1837 (3)	0.2294 (2)	0.5641 (2)	0.0186 (4)	
H1	−0.2525	0.2675	0.5294	0.022*	
N2	0.1383 (3)	0.0420 (2)	0.7154 (2)	0.0210 (5)	

C1	0.3775 (3)	0.4724 (2)	0.3230 (2)	0.0151 (5)
C2	0.4409 (3)	0.5295 (3)	0.2416 (2)	0.0184 (5)
H2	0.5368	0.5838	0.2658	0.022*
C3	0.3651 (3)	0.5080 (3)	0.1248 (3)	0.0229 (6)
H3	0.4078	0.5482	0.0694	0.027*
C4	0.2268 (3)	0.4272 (3)	0.0906 (2)	0.0240 (6)
C5	0.1589 (3)	0.3723 (3)	0.1713 (3)	0.0242 (6)
H5	0.0623	0.3193	0.1472	0.029*
C6	0.2349 (3)	0.3965 (3)	0.2886 (2)	0.0194 (5)
H6	0.1891	0.3609	0.3457	0.023*
C7	−0.2277 (3)	0.1231 (3)	0.6020 (3)	0.0202 (5)
H7	−0.3331	0.0915	0.5928	0.024*
C8	−0.1239 (3)	0.0602 (3)	0.6534 (2)	0.0188 (5)
H8	−0.1573	−0.0146	0.6797	0.023*
C9	0.0341 (3)	0.1054 (3)	0.6681 (2)	0.0159 (5)
C10	0.0741 (3)	0.2198 (3)	0.6290 (2)	0.0173 (5)
H10	0.1781	0.2558	0.6383	0.021*
C11	−0.0357 (3)	0.2780 (3)	0.5785 (2)	0.0182 (5)
H11	−0.0073	0.3545	0.5528	0.022*
C12	0.3025 (3)	0.0848 (3)	0.7299 (3)	0.0281 (6)
H12A	0.3274	0.1224	0.6648	0.042*
H12B	0.3610	0.0023	0.7212	0.042*
H12C	0.3289	0.1583	0.8134	0.042*
C13	0.0909 (4)	−0.0632 (3)	0.7687 (3)	0.0308 (7)
H13A	0.0393	−0.0169	0.8453	0.046*
H13B	0.1812	−0.1062	0.7896	0.046*
H13C	0.0204	−0.1371	0.7070	0.046*
C14	0.5185 (4)	0.1403 (3)	1.0834 (3)	0.0282 (6)
H14	0.5317	0.2364	1.1396	0.034*
C15	0.6185 (4)	0.0876 (3)	0.9901 (3)	0.0249 (6)
C16	0.5999 (4)	−0.0512 (3)	0.9068 (3)	0.0276 (6)
H16	0.6685	−0.0853	0.8429	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01253 (12)	0.01045 (11)	0.01205 (11)	0.00037 (8)	−0.00207 (8)	0.00356 (8)
Br1	0.01696 (13)	0.01231 (11)	0.02127 (13)	−0.00079 (9)	−0.00159 (9)	0.00599 (9)
Br2	0.01604 (13)	0.01762 (12)	0.01965 (13)	0.00199 (9)	0.00089 (9)	0.00756 (10)
Br3	0.0333 (2)	0.0299 (2)	0.0258 (2)	−0.00401 (17)	−0.00423 (17)	0.01252 (17)
Cl2	0.0333 (2)	0.0299 (2)	0.0258 (2)	−0.00401 (17)	−0.00423 (17)	0.01252 (17)
Cl1	0.0435 (5)	0.0421 (4)	0.0144 (3)	0.0164 (3)	−0.0097 (3)	0.0016 (3)
N1	0.0168 (11)	0.0179 (10)	0.0218 (11)	0.0034 (8)	−0.0034 (9)	0.0087 (9)
N2	0.0219 (12)	0.0216 (11)	0.0209 (11)	0.0057 (9)	−0.0017 (9)	0.0094 (9)
C1	0.0183 (12)	0.0117 (10)	0.0125 (11)	0.0033 (9)	−0.0015 (9)	0.0012 (9)
C2	0.0183 (12)	0.0189 (12)	0.0178 (12)	0.0046 (10)	0.0002 (10)	0.0063 (10)
C3	0.0261 (14)	0.0282 (14)	0.0164 (12)	0.0111 (11)	0.0038 (11)	0.0092 (11)
C4	0.0275 (15)	0.0252 (13)	0.0137 (12)	0.0118 (11)	−0.0050 (10)	0.0003 (10)

C5	0.0222 (14)	0.0218 (13)	0.0231 (14)	0.0012 (11)	−0.0085 (11)	0.0032 (11)
C6	0.0188 (13)	0.0193 (12)	0.0179 (12)	−0.0006 (10)	−0.0035 (10)	0.0052 (10)
C7	0.0176 (13)	0.0194 (12)	0.0216 (13)	−0.0016 (10)	−0.0003 (10)	0.0060 (10)
C8	0.0229 (14)	0.0158 (11)	0.0178 (12)	−0.0011 (10)	0.0010 (10)	0.0066 (10)
C9	0.0206 (13)	0.0141 (11)	0.0114 (11)	0.0054 (9)	0.0000 (9)	0.0025 (9)
C10	0.0165 (12)	0.0172 (11)	0.0187 (12)	−0.0010 (9)	0.0004 (10)	0.0075 (10)
C11	0.0209 (13)	0.0161 (11)	0.0181 (12)	−0.0012 (10)	−0.0009 (10)	0.0075 (10)
C12	0.0196 (14)	0.0363 (16)	0.0294 (15)	0.0120 (12)	−0.0009 (12)	0.0124 (13)
C13	0.0401 (18)	0.0283 (15)	0.0313 (16)	0.0090 (13)	−0.0012 (13)	0.0192 (13)
C14	0.0389 (17)	0.0190 (12)	0.0208 (14)	0.0096 (12)	−0.0056 (12)	0.0004 (10)
C15	0.0312 (15)	0.0216 (13)	0.0192 (13)	0.0061 (11)	−0.0089 (11)	0.0055 (11)
C16	0.0338 (16)	0.0253 (14)	0.0197 (13)	0.0110 (12)	−0.0019 (12)	0.0028 (11)

Geometric parameters (Å, °)

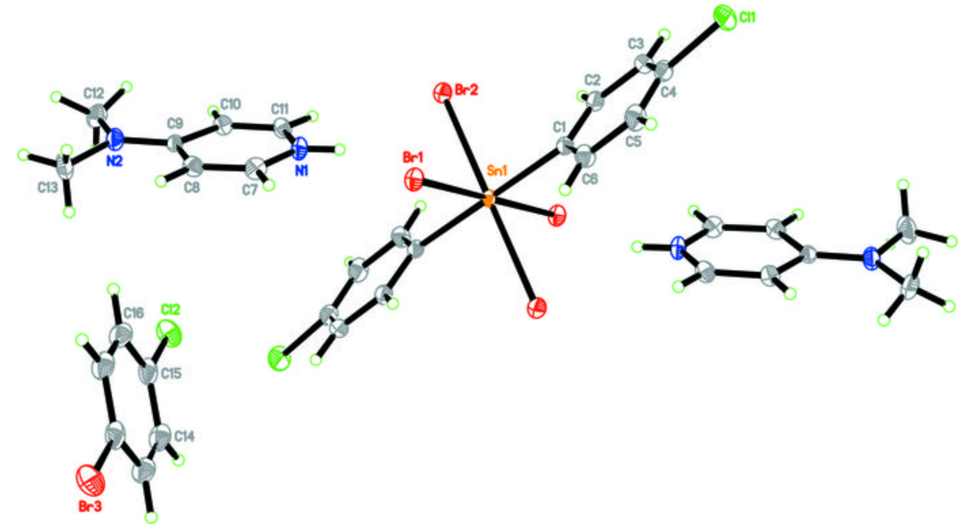
Sn1—C1^{i}	2.148 (3)	C5—H5	0.9500
Sn1—C1	2.148 (3)	C6—H6	0.9500
Sn1—Br2^{i}	2.7172 (9)	C7—C8	1.357 (4)
Sn1—Br2	2.7172 (8)	C7—H7	0.9500
Sn1—Br1	2.7319 (7)	C8—C9	1.418 (4)
Sn1—Br1^{i}	2.7319 (7)	C8—H8	0.9500
Br3—C15	1.807 (3)	C9—C10	1.420 (3)
Cl1—C4	1.744 (3)	C10—C11	1.357 (4)
N1—C11	1.344 (3)	C10—H10	0.9500
N1—C7	1.346 (3)	C11—H11	0.9500
N1—H1	0.8800	C12—H12A	0.9800
N2—C9	1.337 (3)	C12—H12B	0.9800
N2—C13	1.460 (4)	C12—H12C	0.9800
N2—C12	1.465 (4)	C13—H13A	0.9800
C1—C2	1.386 (4)	C13—H13B	0.9800
C1—C6	1.392 (4)	C13—H13C	0.9800
C2—C3	1.392 (4)	C14—C16^{ii}	1.378 (5)
C2—H2	0.9500	C14—C15	1.389 (4)
C3—C4	1.382 (4)	C14—H14	0.9500
C3—H3	0.9500	C15—C16	1.379 (4)
C4—C5	1.383 (4)	C16—C14^{ii}	1.378 (5)
C5—C6	1.391 (4)	C16—H16	0.9500
C1^{i}—Sn1—C1	180.0	C1—C6—H6	119.7
C1^{i}—Sn1—Br2^{i}	89.62 (7)	N1—C7—C8	121.0 (2)
C1—Sn1—Br2^{i}	90.38 (7)	N1—C7—H7	119.5
C1^{i}—Sn1—Br2	90.38 (7)	C8—C7—H7	119.5
C1—Sn1—Br2	89.62 (7)	C7—C8—C9	120.5 (2)
Br2^{i}—Sn1—Br2	180.0	C7—C8—H8	119.8
C1^{i}—Sn1—Br1	89.88 (7)	C9—C8—H8	119.8
C1—Sn1—Br1	90.12 (7)	N2—C9—C8	121.3 (2)
Br2^{i}—Sn1—Br1	91.55 (3)	N2—C9—C10	122.5 (2)

Br2—Sn1—Br1	88.45 (3)	C8—C9—C10	116.2 (2)
C1^{i}—Sn1—Br1^{i}	90.12 (7)	C11—C10—C9	120.2 (2)
C1—Sn1—Br1^{i}	89.88 (7)	C11—C10—H10	119.9
Br2^{i}—Sn1—Br1^{i}	88.45 (3)	C9—C10—H10	119.9
Br2—Sn1—Br1^{i}	91.55 (3)	N1—C11—C10	121.3 (2)
Br1—Sn1—Br1^{i}	180.0	N1—C11—H11	119.4
C11—N1—C7	120.7 (2)	C10—C11—H11	119.4
C11—N1—H1	119.6	N2—C12—H12A	109.5
C7—N1—H1	119.6	N2—C12—H12B	109.5
C9—N2—C13	120.7 (2)	H12A—C12—H12B	109.5
C9—N2—C12	122.5 (2)	N2—C12—H12C	109.5
C13—N2—C12	116.4 (2)	H12A—C12—H12C	109.5
C2—C1—C6	119.5 (2)	H12B—C12—H12C	109.5
C2—C1—Sn1	120.03 (18)	N2—C13—H13A	109.5
C6—C1—Sn1	120.51 (19)	N2—C13—H13B	109.5
C1—C2—C3	120.5 (3)	H13A—C13—H13B	109.5
C1—C2—H2	119.7	N2—C13—H13C	109.5
C3—C2—H2	119.7	H13A—C13—H13C	109.5
C4—C3—C2	118.9 (3)	H13B—C13—H13C	109.5
C4—C3—H3	120.6	C16^{ii}—C14—C15	119.1 (3)
C2—C3—H3	120.6	C16^{ii}—C14—H14	120.4
C3—C4—C5	121.8 (3)	C15—C14—H14	120.4
C3—C4—Cl1	118.7 (2)	C16—C15—C14	121.1 (3)
C5—C4—Cl1	119.5 (2)	C16—C15—Br3	119.9 (2)
C4—C5—C6	118.6 (3)	C14—C15—Br3	119.0 (2)
C4—C5—H5	120.7	C14^{ii}—C16—C15	119.7 (3)
C6—C5—H5	120.7	C14^{ii}—C16—H16	120.1
C5—C6—C1	120.6 (3)	C15—C16—H16	120.1
C5—C6—H6	119.7		
C1^{i}—Sn1—C1—C2	19 (100)	C2—C1—C6—C5	2.8 (4)
Br2^{i}—Sn1—C1—C2	134.73 (19)	Sn1—C1—C6—C5	−176.7 (2)
Br2—Sn1—C1—C2	−45.27 (19)	C11—N1—C7—C8	−1.4 (4)
Br1—Sn1—C1—C2	−133.72 (19)	N1—C7—C8—C9	0.0 (4)
Br1^{i}—Sn1—C1—C2	46.28 (19)	C13—N2—C9—C8	−8.1 (4)
C1^{i}—Sn1—C1—C6	−161 (100)	C12—N2—C9—C8	179.0 (2)
Br2^{i}—Sn1—C1—C6	−45.7 (2)	C13—N2—C9—C10	172.4 (2)
Br2—Sn1—C1—C6	134.3 (2)	C12—N2—C9—C10	−0.5 (4)
Br1—Sn1—C1—C6	45.8 (2)	C7—C8—C9—N2	−178.2 (2)
Br1^{i}—Sn1—C1—C6	−134.2 (2)	C7—C8—C9—C10	1.3 (4)
C6—C1—C2—C3	−1.9 (4)	N2—C9—C10—C11	178.2 (2)
Sn1—C1—C2—C3	177.68 (19)	C8—C9—C10—C11	−1.3 (4)
C1—C2—C3—C4	−0.9 (4)	C7—N1—C11—C10	1.4 (4)
C2—C3—C4—C5	2.8 (4)	C9—C10—C11—N1	0.0 (4)
C2—C3—C4—Cl1	−175.6 (2)	C16^{ii}—C14—C15—C16	0.7 (5)
C3—C4—C5—C6	−1.8 (4)	C16^{ii}—C14—C15—Br3	179.7 (2)

Cl1—C4—C5—C6	176.5 (2)	C14—C15—C16—C14^{ii}	−0.7 (5)
C4—C5—C6—C1	−1.0 (4)	Br3—C15—C16—C14^{ii}	−179.7 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+2.

Fig. 1


Cl1
C3
Br2
C2
C12
C10
C11
C4
C9
C1
C5
N2
N1
Br1
Sn1
C13
C8
C7
C6
C16
Cl2
C15
C14
Br3