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

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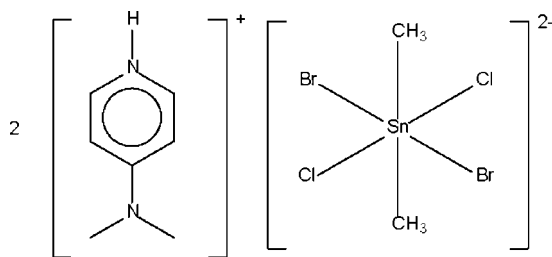
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; disorder in main residue; R factor = 0.046; wR factor = 0.124; data-to-parameter ratio = 22.6.

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) Å³ $Z = 1$ Mo $K\alpha$ radiation $\mu = 4.72$ mm⁻¹ $T = 100$ (2) K

0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer

Absorption 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

 $\Delta\rho_{\text{max}} = 0.82$ e Å⁻³ $\Delta\rho_{\text{min}} = -1.14$ e Å⁻³

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 ⁱ	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: publCIF (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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supplementary materials

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

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

K. M. Lo and S. W. Ng

Comment

Bis(4-methylaminopyridinium) tetrabromidodiphenylstannate is produced from the cleavage of the mixed alkyl/triarylstannate, 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^{IV} 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.

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.

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.

Figures

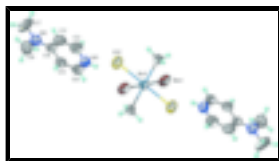


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of $[\text{C}_7\text{H}_{11}\text{N}]_2 [\text{SnBr}_2\text{Cl}_2(\text{CH}_3)_2]$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The disorder is not shown.

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

Crystal data

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

$Z = 1$

supplementary materials

$M_r = 625.83$	$F_{000} = 306$
Triclinic, $P\bar{1}$	$D_x = 1.767 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 7.3573 (3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.7717 (3) \text{ \AA}$	Cell parameters from 1302 reflections
$c = 9.6644 (4) \text{ \AA}$	$\theta = 2.3\text{--}23.1^\circ$
$\alpha = 97.183 (3)^\circ$	$\mu = 4.72 \text{ mm}^{-1}$
$\beta = 107.990 (3)^\circ$	$T = 100 (2) \text{ K}$
$\gamma = 90.052 (2)^\circ$	Prism, colorless
$V = 588.04 (4) \text{ \AA}^3$	$0.25 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer	2756 independent reflections
Radiation source: fine-focus sealed tube	1656 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.045$
$T = 100(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: Multi-scan (SADABS;Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.385, T_{\text{max}} = 0.538$	$k = -11 \rightarrow 11$
4965 measured reflections	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.051P)^2]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2756 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
122 parameters	$\Delta\rho_{\text{max}} = 0.82 \text{ e \AA}^{-3}$
4 restraints	$\Delta\rho_{\text{min}} = -1.14 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	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 (\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 (\AA , $^\circ$)

Sn1—C1 ⁱ	2.225 (5)	C1—H1C	0.9800
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Sn1—C1	2.225 (5)	C2—C3	1.381 (8)
Sn1—Br1	2.690 (1)	C2—H2	0.9500
Sn1—Cl1 ⁱ	2.690 (1)	C3—C4	1.420 (9)
Sn1—Br1 ⁱ	2.690 (1)	C3—H3	0.9500
Sn1—Br2 ⁱ	2.6926 (8)	C4—C5	1.409 (9)
Sn1—Br2	2.6926 (8)	C5—C6	1.369 (8)
Sn1—Cl2 ⁱ	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 ⁱ —Sn1—C1	180.0	Sn1—C1—H1A	109.5
C1 ⁱ —Sn1—Br1	88.38 (14)	Sn1—C1—H1B	109.5
C1—Sn1—Br1	91.62 (14)	H1A—C1—H1B	109.5
C1 ⁱ —Sn1—Cl1 ⁱ	91.62 (14)	Sn1—C1—H1C	109.5
C1—Sn1—Cl1 ⁱ	88.38 (14)	H1A—C1—H1C	109.5
Br1—Sn1—Cl1 ⁱ	180.0	H1B—C1—H1C	109.5
C1 ⁱ —Sn1—Br1 ⁱ	91.62 (14)	N1—C2—C3	121.1 (6)
C1—Sn1—Br1 ⁱ	88.38 (14)	N1—C2—H2	119.5
Br1—Sn1—Br1 ⁱ	180.0	C3—C2—H2	119.5
Cl1 ⁱ —Sn1—Br1 ⁱ	0.0	C2—C3—C4	120.0 (6)
C1 ⁱ —Sn1—Br2 ⁱ	89.83 (13)	C2—C3—H3	120.0
C1—Sn1—Br2 ⁱ	90.17 (13)	C4—C3—H3	120.0
Br1—Sn1—Br2 ⁱ	89.12 (3)	N2—C4—C5	122.5 (6)
Cl1 ⁱ —Sn1—Br2 ⁱ	90.88 (3)	N2—C4—C3	121.6 (6)
Br1 ⁱ —Sn1—Br2 ⁱ	90.88 (3)	C5—C4—C3	115.9 (6)
C1 ⁱ —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 ⁱ —Sn1—Br2	89.12 (3)	N1—C6—C5	120.4 (6)
Br1 ⁱ —Sn1—Br2	89.12 (3)	N1—C6—H6	119.8
Br2 ⁱ —Sn1—Br2	180.0	C5—C6—H6	119.8
C1 ⁱ —Sn1—Cl2 ⁱ	89.83 (13)	N2—C7—H7A	109.5
C1—Sn1—Cl2 ⁱ	90.17 (13)	N2—C7—H7B	109.5
Br1—Sn1—Cl2 ⁱ	89.12 (3)	H7A—C7—H7B	109.5
Cl1 ⁱ —Sn1—Cl2 ⁱ	90.88 (3)	N2—C7—H7C	109.5
Br1 ⁱ —Sn1—Cl2 ⁱ	90.88 (3)	H7A—C7—H7C	109.5
Br2 ⁱ —Sn1—Cl2 ⁱ	0.000 (13)	H7B—C7—H7C	109.5
Br2—Sn1—Cl2 ⁱ	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 codes: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

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

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

