

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

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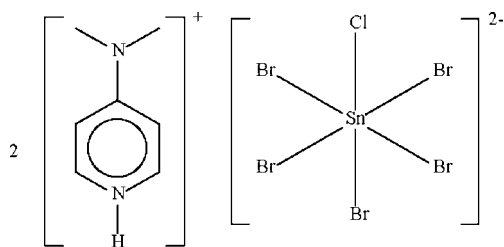
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.021; wR factor = 0.053; data-to-parameter ratio = 21.6.

In the title compound, $(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_5\text{Cl}]$, there is Br/Cl disorder in 0.6561 (12):0.3439 (12) and 0.8438 (12):0.1561 (12) ratios over two of three halide sites in the centrosymmetric anion, such that an overall formulation of $[\text{SnBr}_5\text{Cl}]^{2-}$ arises. In the crystal, associations of two cations and one anion linked by $\text{N}-\text{H}\cdots\text{Br}$ hydrogen bonds occur.

Related literature

For related 4-dimethylaminopyridinium halogenoorganostannates, see: Lo & Ng (2008); Norhafiza *et al.* (2008); Yau *et al.* (2008).



Experimental

Crystal data

$(\text{C}_7\text{H}_{11}\text{N}_2)_2[\text{SnBr}_5\text{Cl}]$
 $M_r = 800.05$
Monoclinic, $P2_1/c$
 $a = 8.4424$ (1) Å
 $b = 11.8821$ (2) Å

$c = 11.8868$ (2) Å
 $\beta = 107.123$ (1)°
 $V = 1139.55$ (3) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 10.01$ mm⁻¹
 $T = 100$ K

0.30 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.152$, $T_{\max} = 0.434$
(expected range = 0.129–0.367)

9261 measured reflections
2613 independent reflections
2408 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$
 $wR(F^2) = 0.053$
 $S = 1.01$
2613 reflections
121 parameters

4 restraints
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.91$ e Å⁻³
 $\Delta\rho_{\min} = -0.95$ e Å⁻³

Table 1

Selected bond lengths (Å). $X = (\text{Br}, \text{Cl})$.

| | | | |
|--------|------------|--------|------------|
| Sn1—X1 | 2.5608 (3) | Sn1—X3 | 2.5687 (3) |
| Sn1—X2 | 2.5618 (3) | | |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---------------------------------------|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{Br1}$ | 0.88 | 2.47 | 3.327 (2) | 165 |

Data collection: APEX2 software (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).

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: HB2965).

References

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supplementary materials

Acta Cryst. (2009). E65, m645 [doi:10.1107/S160053680901705X]

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

Y. Jang, 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 3 hours. Colourless blocks of (I) separated from the cool solution after a day. The crystal structure showed that the benzyl groups on tin had been cleaved in the reaction.

Refinement

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

Two of the three halogen atoms in the stannate are disordered. The pair of Br1/Cl1 and Br2/Cl2 atoms initially refined to nearly 1.5Br and 0.5Cl atoms; the total occupancy of the disordered bromine atoms was then fixed as exactly 1.5. The occupancy of the disordered chlorine atoms was similarly set to be exactly 0.5.

The U^{ij} values of the Br1 and Cl1 atoms were restrained to be identical, as were those of the Br2 and Cl2 atoms.

Figures

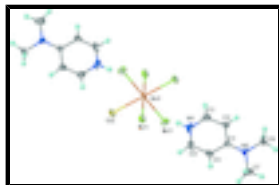


Fig. 1. The molecular structure of (I) viewed at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

(C₇H₁₁N₂)₂[SnBr₅Cl]

$M_r = 800.05$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.4424 (1) \text{ \AA}$

$b = 11.8821 (2) \text{ \AA}$

$c = 11.8868 (2) \text{ \AA}$

$\beta = 107.123 (1)^\circ$

$V = 1139.55 (3) \text{ \AA}^3$

$F_{000} = 752$

$D_x = 2.332 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 5777 reflections

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

$\mu = 10.01 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, colourless

$0.30 \times 0.10 \times 0.10 \text{ mm}$

supplementary materials

$Z = 2$

Data collection

| | |
|---|--|
| Bruker SMART APEX CCD diffractometer | 2613 independent reflections |
| Radiation source: fine-focus sealed tube | 2408 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.022$ |
| $T = 100$ K | $\theta_{\text{max}} = 27.5^\circ$ |
| ω scans | $\theta_{\text{min}} = 2.5^\circ$ |
| Absorption correction: Multi-scan (SADABS; Sheldrick, 1996) | $h = -10 \rightarrow 10$ |
| $T_{\text{min}} = 0.152$, $T_{\text{max}} = 0.434$ | $k = -15 \rightarrow 15$ |
| 9261 measured reflections | $l = -15 \rightarrow 15$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.021$ | H-atom parameters constrained |
| $wR(F^2) = 0.053$ | $w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 1.7721P]$ |
| $S = 1.01$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2613 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 121 parameters | $\Delta\rho_{\text{max}} = 0.91 \text{ e } \text{\AA}^{-3}$ |
| 4 restraints | $\Delta\rho_{\text{min}} = -0.95 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|--------------|-------------|----------------------------------|-------------|
| Sn1 | 0.5000 | 0.5000 | 0.5000 | 0.01035 (7) | |
| Br1 | 0.50979 (5) | 0.63602 (3) | 0.66902 (3) | 0.02104 (10) | 0.6561 (12) |
| Br2 | 0.58402 (4) | 0.33881 (2) | 0.64884 (3) | 0.02209 (9) | 0.8438 (12) |
| Br3 | 0.80725 (3) | 0.53820 (3) | 0.52248 (3) | 0.02568 (8) | |
| Cl1 | 0.50979 (5) | 0.63602 (3) | 0.66902 (3) | 0.02104 (10) | 0.3439 (12) |
| Cl2 | 0.58402 (4) | 0.33881 (2) | 0.64884 (3) | 0.02209 (9) | 0.1561 (12) |
| N1 | 0.6520 (3) | 0.8746 (2) | 0.5886 (3) | 0.0271 (6) | |
| H1 | 0.5999 | 0.8123 | 0.5962 | 0.032* | |
| N2 | 0.9139 (3) | 1.15682 (19) | 0.5545 (2) | 0.0186 (5) | |
| C1 | 0.8253 (3) | 1.0669 (2) | 0.5659 (2) | 0.0154 (5) | |
| C2 | 0.7373 (4) | 1.0025 (2) | 0.4666 (3) | 0.0194 (5) | |
| H2 | 0.7358 | 1.0257 | 0.3898 | 0.023* | |
| C3 | 0.6551 (4) | 0.9077 (2) | 0.4809 (3) | 0.0245 (6) | |
| H3 | 0.5991 | 0.8642 | 0.4139 | 0.029* | |
| C4 | 0.7277 (4) | 0.9358 (3) | 0.6848 (3) | 0.0251 (6) | |

| | | | | |
|-----|------------|------------|------------|------------|
| H4 | 0.7203 | 0.9125 | 0.7595 | 0.030* |
| C5 | 0.8137 (4) | 1.0296 (2) | 0.6768 (3) | 0.0211 (6) |
| H5 | 0.8670 | 1.0711 | 0.7459 | 0.025* |
| C6 | 0.9127 (4) | 1.1990 (3) | 0.4393 (3) | 0.0249 (6) |
| H6A | 0.9488 | 1.1393 | 0.3955 | 0.037* |
| H6B | 0.9883 | 1.2633 | 0.4490 | 0.037* |
| H6C | 0.8002 | 1.2229 | 0.3958 | 0.037* |
| C7 | 1.0105 (4) | 1.2208 (3) | 0.6571 (3) | 0.0291 (7) |
| H7A | 0.9353 | 1.2650 | 0.6887 | 0.044* |
| H7B | 1.0866 | 1.2715 | 0.6336 | 0.044* |
| H7C | 1.0740 | 1.1687 | 0.7175 | 0.044* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Sn1 | 0.00978 (11) | 0.01195 (11) | 0.00936 (12) | -0.00163 (8) | 0.00287 (9) | -0.00006 (8) |
| Br1 | 0.0306 (2) | 0.01735 (16) | 0.01761 (19) | -0.00301 (13) | 0.01094 (15) | -0.00399 (12) |
| Br2 | 0.03025 (18) | 0.01759 (15) | 0.01582 (17) | -0.00058 (12) | 0.00271 (13) | 0.00430 (11) |
| Br3 | 0.01302 (14) | 0.03789 (17) | 0.02652 (17) | -0.00656 (11) | 0.00643 (12) | -0.00107 (12) |
| Cl1 | 0.0306 (2) | 0.01735 (16) | 0.01761 (19) | -0.00301 (13) | 0.01094 (15) | -0.00399 (12) |
| Cl2 | 0.03025 (18) | 0.01759 (15) | 0.01582 (17) | -0.00058 (12) | 0.00271 (13) | 0.00430 (11) |
| N1 | 0.0210 (12) | 0.0210 (12) | 0.0407 (17) | -0.0003 (10) | 0.0115 (11) | 0.0092 (11) |
| N2 | 0.0175 (11) | 0.0195 (11) | 0.0167 (12) | -0.0033 (9) | 0.0019 (9) | -0.0018 (9) |
| C1 | 0.0123 (12) | 0.0183 (12) | 0.0145 (13) | 0.0038 (9) | 0.0025 (10) | 0.0005 (10) |
| C2 | 0.0183 (13) | 0.0212 (13) | 0.0170 (14) | 0.0007 (10) | 0.0024 (11) | -0.0009 (11) |
| C3 | 0.0208 (14) | 0.0230 (14) | 0.0265 (16) | -0.0016 (11) | 0.0019 (12) | -0.0023 (12) |
| C4 | 0.0214 (14) | 0.0320 (15) | 0.0256 (16) | 0.0108 (12) | 0.0130 (12) | 0.0122 (13) |
| C5 | 0.0207 (14) | 0.0283 (14) | 0.0141 (14) | 0.0061 (11) | 0.0049 (11) | 0.0018 (11) |
| C6 | 0.0240 (14) | 0.0240 (14) | 0.0251 (16) | -0.0053 (11) | 0.0047 (12) | 0.0069 (12) |
| C7 | 0.0290 (16) | 0.0304 (16) | 0.0233 (17) | -0.0097 (12) | 0.0007 (13) | -0.0110 (13) |

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

| | | | |
|----------------------|------------|--------|-----------|
| Sn1—Br1 | 2.5608 (3) | C1—C2 | 1.419 (4) |
| Sn1—Cl1 ⁱ | 2.5608 (3) | C1—C5 | 1.421 (4) |
| Sn1—Br1 ⁱ | 2.5608 (3) | C2—C3 | 1.360 (4) |
| Sn1—Br2 | 2.5618 (3) | C2—H2 | 0.9500 |
| Sn1—Cl2 ⁱ | 2.5618 (3) | C3—H3 | 0.9500 |
| Sn1—Br2 ⁱ | 2.5618 (3) | C4—C5 | 1.348 (4) |
| Sn1—Br3 ⁱ | 2.5687 (3) | C4—H4 | 0.9500 |
| Sn1—Br3 | 2.5687 (3) | C5—H5 | 0.9500 |
| N1—C4 | 1.347 (4) | C6—H6A | 0.9800 |
| N1—C3 | 1.347 (4) | C6—H6B | 0.9800 |
| N1—H1 | 0.8800 | C6—H6C | 0.9800 |
| N2—C1 | 1.334 (3) | C7—H7A | 0.9800 |
| N2—C6 | 1.456 (4) | C7—H7B | 0.9800 |
| N2—C7 | 1.464 (4) | C7—H7C | 0.9800 |

supplementary materials

| | | | |
|--|--------------|-------------|------------|
| Br1—Sn1—Cl1 ⁱ | 180.0 | C1—N2—C6 | 121.5 (2) |
| Br1—Sn1—Br1 ⁱ | 180.0 | C1—N2—C7 | 121.6 (2) |
| Cl1 ⁱ —Sn1—Br1 ⁱ | 0.000 (11) | C6—N2—C7 | 116.9 (2) |
| Br1—Sn1—Br2 | 89.520 (11) | N2—C1—C2 | 121.3 (3) |
| Cl1 ⁱ —Sn1—Br2 | 90.480 (11) | N2—C1—C5 | 122.5 (3) |
| Br1 ⁱ —Sn1—Br2 | 90.480 (11) | C2—C1—C5 | 116.2 (3) |
| Br1—Sn1—Cl2 ⁱ | 90.480 (11) | C3—C2—C1 | 120.3 (3) |
| Cl1 ⁱ —Sn1—Cl2 ⁱ | 89.520 (11) | C3—C2—H2 | 119.8 |
| Br1 ⁱ —Sn1—Cl2 ⁱ | 89.520 (11) | C1—C2—H2 | 119.8 |
| Br2—Sn1—Cl2 ⁱ | 180.000 (12) | N1—C3—C2 | 120.9 (3) |
| Br1—Sn1—Br2 ⁱ | 90.480 (11) | N1—C3—H3 | 119.6 |
| Cl1 ⁱ —Sn1—Br2 ⁱ | 89.520 (11) | C2—C3—H3 | 119.6 |
| Br1 ⁱ —Sn1—Br2 ⁱ | 89.520 (11) | N1—C4—C5 | 121.1 (3) |
| Br2—Sn1—Br2 ⁱ | 180.000 (12) | N1—C4—H4 | 119.4 |
| Cl2 ⁱ —Sn1—Br2 ⁱ | 0.00 (2) | C5—C4—H4 | 119.4 |
| Br1—Sn1—Br3 ⁱ | 89.512 (11) | C4—C5—C1 | 120.6 (3) |
| Cl1 ⁱ —Sn1—Br3 ⁱ | 90.488 (11) | C4—C5—H5 | 119.7 |
| Br1 ⁱ —Sn1—Br3 ⁱ | 90.488 (11) | C1—C5—H5 | 119.7 |
| Br2—Sn1—Br3 ⁱ | 90.316 (10) | N2—C6—H6A | 109.5 |
| Cl2 ⁱ —Sn1—Br3 ⁱ | 89.684 (10) | N2—C6—H6B | 109.5 |
| Br2 ⁱ —Sn1—Br3 ⁱ | 89.684 (10) | H6A—C6—H6B | 109.5 |
| Br1—Sn1—Br3 | 90.488 (11) | N2—C6—H6C | 109.5 |
| Cl1 ⁱ —Sn1—Br3 | 89.512 (11) | H6A—C6—H6C | 109.5 |
| Br1 ⁱ —Sn1—Br3 | 89.512 (11) | H6B—C6—H6C | 109.5 |
| Br2—Sn1—Br3 | 89.684 (10) | N2—C7—H7A | 109.5 |
| Cl2 ⁱ —Sn1—Br3 | 90.316 (10) | N2—C7—H7B | 109.5 |
| Br2 ⁱ —Sn1—Br3 | 90.316 (10) | H7A—C7—H7B | 109.5 |
| Br3 ⁱ —Sn1—Br3 | 180.0 | N2—C7—H7C | 109.5 |
| C4—N1—C3 | 120.8 (3) | H7A—C7—H7C | 109.5 |
| C4—N1—H1 | 119.6 | H7B—C7—H7C | 109.5 |
| C3—N1—H1 | 119.6 | | |
| C6—N2—C1—C2 | 5.6 (4) | C4—N1—C3—C2 | 1.3 (4) |
| C7—N2—C1—C2 | -177.4 (3) | C1—C2—C3—N1 | 1.7 (4) |
| C6—N2—C1—C5 | -174.8 (3) | C3—N1—C4—C5 | -2.4 (4) |
| C7—N2—C1—C5 | 2.2 (4) | N1—C4—C5—C1 | 0.6 (4) |
| N2—C1—C2—C3 | 176.4 (3) | N2—C1—C5—C4 | -177.5 (3) |
| C5—C1—C2—C3 | -3.3 (4) | C2—C1—C5—C4 | 2.2 (4) |

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 | 2.47 | 3.327 (2) | 165 |

Fig. 1

