4-Amidinopyridinium hexachloridostannate(IV) dihydrate

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In the title hydrated molecular salt [systematic name: 4-[amino(iminiumyl)-methyl]pyridin-1-ium hexachloridostannate(IV) dihydrate], (C6H9N3)[SnCl6]2H2O, the tin atom lies on a crystallographic inversion centre and the organic cation shows whole-molecule disorder. Numerous N—H···Cl and O—H···Cl hydrogen bonds link the components in the crystal.

Structure description

The title hydrated molecular salt, with formula (C6H9N3)[SnCl6]2H2O, crystallizes in the triclinic space group P1. The asymmetric unit is constituted by a Sn0.5Cl3 fragment (Sn site symmetry 1), a 4-amidinopyridinium cation (twice protonated at N1 and N2) and a water molecule, as shown in Fig. 1.

The cation shows whole-molecule disorder about an inversion centre and the water molecule is disordered over adjacent positions (O···O = 1.13 Å) and there is also static disorder of two of the chloride ions of the anion. With the exception of Cl3, where the occupancy ratio is 0.67/0.33 (for Cl3A/Cl3B), each disordered atom is shared between two crystallographic sites with occupancies of 0.50. There are no abnormalities in the bond lengths and angles and they are comparable to those of similar types (Liu et al., 2011; Ghallab et al., 2020).

In the extended structure, cationic and anionic layers occur, with water molecules intercalating between them as shown in the projection of the structure onto the ac and bc planes (Figs. 2 and 3). Cohesion in the crystal is ensured by numerous hydrogen bonds (Table 1).
Projection of the crystal packing on the $bc$ plane.

**Synthesis and crystallization**

Following the method of preparation described in the literature (Bouchene et al., 2018), the compound was synthesized via the aqueous technique. A millimeter-sized transparent crystal was formed after three months of slow evaporation at ambient temperature.

**Figure 1**
The molecular structure showing 30% displacement ellipsoids.

**Figure 2**
Projection of the crystal packing on the $ac$ plane.

**Figure 3**
Projection of the crystal packing on the $bc$ plane.

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### Table 1

| Hydrogen-bond geometry ($\AA$, °) |
|---|
| $D$—H···$A$ | $D$—H | $D$···$A$ | $D$—H···$A$ |
| N1—H1···O1WA$^i$ | 0.86 | 1.96 | 2.760 (15) | 154 |
| N1—H1···O1WB$^b$ | 0.86 | 1.87 | 2.649 (15) | 149 |
| N2—H2A···Cl2$^a$ | 0.86 | 2.68 | 3.431 (11) | 147 |
| N3—H3A···O1WA$^{ii}$ | 0.86 | 2.13 | 2.961 (16) | 162 |
| N3—H3A···O1WB$^{ii}$ | 0.86 | 1.96 | 2.795 (16) | 163 |
| N3—H3B···Cl3$^{ii}$ | 0.86 | 2.69 | 3.093 (16) | 110 |
| O1WA—H1WA···Cl2$^a$ | 0.85 | 2.77 | 3.415 (8) | 134 |
| O1WA—H1WB···Cl3$^{ii}$ | 0.85 | 2.41 | 3.154 (9) | 147 |
| O1WB—H1WC···ClA$^{i}$ | 0.85 | 2.60 | 3.305 (10) | 142 |
| O1WB—H1WC···ClB$^{i}$ | 0.85 | 2.36 | 3.085 (10) | 144 |
| O1WB—H1WC···ClA$^{ii}$ | 0.85 | 2.69 | 3.251 (12) | 124 |
| O1WB—H1WC···ClB$^{ii}$ | 0.85 | 2.83 | 3.596 (13) | 126 |
| C1—H1A···Cl3A$^{iii}$ | 0.93 | 2.67 | 3.561 (17) | 161 |
| C1—H1A···Cl3B$^{iii}$ | 0.93 | 2.43 | 3.356 (17) | 174 |
| C5—H5···ClA$^{iii}$ | 0.93 | 2.80 | 3.674 (12) | 157 |
| C5—H5···ClB$^{iii}$ | 0.93 | 2.56 | 3.385 (12) | 149 |

Symmetry codes: (i) $x-1, -y+4, z-2+1$; (ii) $x, -y+1, -z+1$; (iii) $x, y-1, z$; (iv) $-x+1, -y+2, z+2$; (v) $x, y+1, z+1$; (vi) $x, y+2, z+1$; (vii) $-x+1, -y+3, -z+2$; (viii) $-x+1, -y+2, -z+1$.

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### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The disordered atoms were treated with constraints on distances and angles (by the SAME command and PART options). With the exception of Cl3, where the ratio is 0.67/0.33, each disordered atom is shared between two crystallographic sites with occupancy rates of 0.50.

**Table 2**

**Experimental details.**

| Crystal data | (C$_6$H$_9$N$_3$)[SnCl$_6$]·2H$_2$O |
|---|---|
| M$_r$ | 490.58 |
| Crystal system, space group | Triclinic, $P\bar{T}$ |
| Temperature (K) | 296 |
| $a$, $b$, $c$ (Å) | 7.4224 (13), 7.4518 (11), 8.4986 (16) |
| $\alpha$, $\beta$, $\gamma$ (°) | 105.726 (7), 97.426 (9), 112.383 (7) |
| V ($Å^3$) | 403.85 (12) |
| Z | 1 |
| Radiation type | Mo Kα |
| $\mu$ (mm$^{-1}$) | 2.57 |
| Crystal size (mm) | 0.17 × 0.13 × 0.11 |

**Data collection**

| Diffractometer | Bruker APEXII CCD |
|---|---|
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) |
| $T_{min}$, $T_{max}$ | 0.676, 0.754 |
| No. of measured, independent and observed $|I|>2\sigma(I)$ reflections | 10469, 2442, 1889 |
| R$_{int}$, (sin $\theta$/λ)$_{max}$ ($Å^{-1}$) | 0.028, 0.714 |

**Refinement**

| $R[F^2>2\sigma(F^2)]$, wR($F^2$), S | 0.046, 0.085, 1.15 |
| No. of reflections | 2442 |
| No. of parameters | 154 |
| No. of restraints | 53 |
| H-atom treatment | H-atom parameters constrained |
| Δρ$_{max}$, Δρ$_{min}$ ($e Å^{-3}$) | 1.22, −1.35 |

Computer programs: APEX2 and SAINT (Bruker, 2016), olex2.solve (Bourhis et al., 2015), SHELXL (Sheldrick, 2015) and OLEX2 (Dolomanov et al., 2009).
Acknowledgements

Thanks are due to DRSDT–Algeria for support.

Funding information

Funding for this research was provided by: Unité de recherche de chimie de l’environnement, moléculaire et structurale UR.CHEMS; Direction Générale de la Recherche Scientifique et du Développement Technologique DGRSDT Algérie.

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full crystallographic data

IUCrData (2022). 7, x220195  [https://doi.org/10.1107/S241431462200195X]

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4-[Amino(iminiumyl)methyl]pyridin-1-ium hexachloridostannate(IV) dihydrate

Crystal data

(C6H9N3)[SnCl6]·2H2O

Z = 1

F(000) = 238

Mr = 490.58

Dm = 2.017 Mg m−3

Triclinic, P T

Mo Kα radiation, λ = 0.71073 Å

a = 7.4224 (13) Å

θ = 5.0–30.5°

b = 7.4518 (11) Å

μ = 2.57 mm−1

c = 8.4986 (16) Å

T = 296 K

α = 105.726 (7)°

Block, colourless

β = 97.426 (9)°

0.17 × 0.13 × 0.11 mm

γ = 112.383 (7)°

V = 403.85 (12) Å³

Data collection

Bruker APEXII CCD

φ and ω scans

2442 independent reflections

Absorption correction: multi-scan

1889 reflections with I > 2σ(I)

(SADABS; Bruker, 2016)

Tmin = 0.676, Tmax = 0.754

10469 measured reflections

l = −12→12

Refinement

Refinement on F²

Primary atom site location: iterative

Least-squares matrix: full

Hydrogen site location: mixed

R[F² > 2σ(F²)] = 0.046

H-atom parameters constrained

wR(F²) = 0.085

w = 1/[σ²(F²̄) + (0.0167P)² + 0.8036P]

where P = (F² + 2F̄²)/3

(Δ/σ)max < 0.001

2442 reflections

Δρmax = 1.22 e Å⁻³

154 parameters

Δρmin = −1.35 e Å⁻³

53 restraints

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.
Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|     | x     | y     | z     | Uiso* / Ueq | Occ. (<1) |
|-----|-------|-------|-------|-------------|-----------|
| Sn1 | 0.000000 | 0.500000 | 0.500000 | 0.04673 (15) |            |
| Cl2 | -0.0563 (2) | 0.39683 (16) | 0.19228 (11) | 0.0675 (4) |            |
| Cl1B| 0.2964 (10) | 0.7987 (8) | 0.5355 (8) | 0.0640 (13) | 0.5       |
| Cl3B| 0.2133 (9) | 0.3209 (8) | 0.5250 (8) | 0.0607 (14) | 0.33      |
| Cl1A| 0.3472 (10) | 0.7539 (8) | 0.5316 (7) | 0.0612 (12) | 0.5       |
| Cl3A| 0.1294 (5) | 0.2493 (4) | 0.4986 (4) | 0.0711 (9) | 0.67      |
| C3  | 0.470 (3) | 1.475 (3) | 0.998 (2) | 0.039 (3) | 0.5       |
| C4  | 0.5060 (13) | 1.6621 (11) | 1.1133 (9) | 0.0462 (19) | 0.5       |
| H4  | 0.465642 | 1.667299 | 1.212925 | 0.055* | 0.5       |
| C5  | 0.5998 (16) | 1.8390 (14) | 1.0820 (13) | 0.062 (2) | 0.5       |
| H5  | 0.618436 | 1.965008 | 1.157590 | 0.074* | 0.5       |
| N1  | 0.666 (2) | 1.8325 (16) | 0.9421 (16) | 0.081 (3) | 0.5       |
| H1  | 0.734961 | 1.946420 | 0.927815 | 0.097* | 0.5       |
| C1  | 0.625 (3) | 1.6494 (18) | 0.8222 (18) | 0.088 (5) | 0.5       |
| H1A | 0.661958 | 1.646634 | 0.721374 | 0.105* | 0.5       |
| C2  | 0.5291 (16) | 1.4684 (15) | 0.8517 (10) | 0.059 (2) | 0.5       |
| H2  | 0.504299 | 1.342167 | 0.772395 | 0.071* | 0.5       |
| C6  | 0.3621 (14) | 1.2750 (14) | 1.0199 (13) | 0.053 (2) | 0.5       |
| N2  | 0.227 (2) | 1.1247 (14) | 0.8868 (15) | 0.099 (4) | 0.5       |
| H2A | 0.153059 | 1.008127 | 0.895028 | 0.119* | 0.5       |
| H2B | 0.211066 | 1.142421 | 0.790976 | 0.119* | 0.5       |
| N3  | 0.397 (2) | 1.2666 (18) | 1.1621 (18) | 0.089 (5) | 0.5       |
| H3A | 0.329233 | 1.154902 | 1.180199 | 0.106* | 0.5       |
| H3B | 0.488756 | 1.372373 | 1.243683 | 0.106* | 0.5       |
| O1WA| 0.0858 (13) | 1.8847 (11) | 1.1795 (10) | 0.069 (2) | 0.5       |
| H1WA| 0.119362 | 1.785736 | 1.167388 | 0.103* | 0.5       |
| H1WB| 0.101422 | 1.945036 | 1.284078 | 0.103* | 0.5       |
| O1WB| 0.2372 (18) | 1.8798 (12) | 1.1997 (9) | 0.091 (3) | 0.5       |
| H1WC| 0.297336 | 1.913349 | 1.302646 | 0.137* | 0.5       |
| H1WD| 0.110355 | 1.828348 | 1.187657 | 0.137* | 0.5       |

Atomic displacement parameters (Å²)

|     | U₁₁   | U₁₂   | U₁₃   | U₂₂   | U₂₃   | U₃₃   |
|-----|-------|-------|-------|-------|-------|-------|
| Sn1 | 0.0750 (3) | 0.02256 (16) | 0.02323 (16) | 0.00466 (17) | 0.00409 (16) | 0.00756 (12) |
| Cl2 | 0.1091 (10) | 0.0438 (5) | 0.0264 (4) | 0.0154 (6) | 0.0077 (5) | 0.0086 (4) |
| Cl1B| 0.064 (3) | 0.046 (2) | 0.0547 (17) | −0.0007 (15) | −0.0018 (17) | 0.0211 (17) |
| Cl3B| 0.067 (4) | 0.051 (3) | 0.056 (2) | 0.025 (2) | −0.004 (2) | 0.019 (2) |
| Cl1A| 0.072 (3) | 0.0429 (19) | 0.0492 (14) | 0.0061 (14) | 0.0161 (18) | 0.0157 (13) |
| Cl3A| 0.111 (3) | 0.0488 (14) | 0.0504 (13) | 0.0290 (14) | 0.0200 (16) | 0.0222 (12) |
| C3  | 0.041 (10) | 0.040 (8) | 0.038 (3) | 0.018 (7) | 0.013 (5) | 0.018 (4) |
| C4  | 0.057 (5) | 0.041 (4) | 0.033 (3) | 0.014 (4) | 0.012 (3) | 0.013 (3) |
| C5  | 0.058 (6) | 0.048 (5) | 0.073 (6) | 0.019 (5) | 0.020 (5) | 0.017 (4) |
| N1  | 0.096 (9) | 0.065 (6) | 0.116 (9) | 0.038 (6) | 0.054 (8) | 0.064 (7) |
| C1  | 0.137 (12) | 0.087 (9) | 0.092 (9) | 0.067 (10) | 0.081 (8) | 0.058 (8) |
|    |     |     |     |     |     |     |     |     |
|----|-----|-----|-----|-----|-----|-----|-----|-----|
| C2 | 0.083 (7) | 0.075 (6) | 0.039 (4) | 0.053 (6) | 0.023 (4) | 0.017 (4) |     |     |
| C6 | 0.049 (6) | 0.045 (4) | 0.064 (6) | 0.019 (4) | 0.018 (5) | 0.020 (4) |     |     |
| N2 | 0.120 (10) | 0.042 (4) | 0.094 (8) | 0.009 (6) | 0.017 (7) | 0.005 (5) |     |     |
| N3 | 0.094 (9) | 0.045 (6) | 0.092 (9) | −0.005 (6) | −0.006 (7) | 0.037 (6) |     |     |
| O1WA | 0.094 (6) | 0.049 (4) | 0.053 (4) | 0.014 (4) | 0.029 (4) | 0.023 (3) |     |     |
| O1WB | 0.133 (8) | 0.045 (4) | 0.044 (4) | −0.006 (5) | −0.005 (5) | 0.018 (3) |     |     |

**Geometric parameters (Å, °)**

| Bond/Angle | Distance/Angle |
|------------|----------------|
| Sn1—Cl2    | 2.4470 (10)    |
| Sn1—Cl2i   | 2.4470 (10)    |
| Sn1—Cl1B   | 2.371 (6)       |
| Sn1—Cl1Bi  | 2.371 (6)       |
| Sn1—Cl1B   | 2.451 (7)       |
| Sn1—Cl3B   | 2.451 (7)       |
| Sn1—Cl1A   | 2.475 (7)       |
| Sn1—Cl1Ai  | 2.475 (7)       |
| Sn1—Cl3A   | 2.402 (4)       |
| Sn1—Cl3A   | 2.402 (4)       |

| Bond/Angle | Distance/Angle |
|------------|----------------|
| Cl2—Sn1—Cl2 | 180.0          |
| Cl2—Sn1—Cl3B | 87.17 (16)     |
| Cl2—Sn1—Cl3Bi | 92.83 (16)    |
| Cl2—Sn1—Cl3B | 92.83 (16)     |
| Cl2—Sn1—Cl3B | 87.17 (16)     |
| Cl2—Sn1—Cl1A | 90.98 (14)     |
| Cl2—Sn1—Cl1A | 89.02 (14)     |
| Cl2—Sn1—Cl1A | 90.98 (14)     |
| Cl2—Sn1—Cl1A | 89.02 (14)     |
| Cl2—Sn1—Cl1A | 89.79 (15)     |
| Cl2—Sn1—Cl1A | 89.79 (15)     |
| Cl2—Sn1—Cl1A | 90.21 (15)     |
| Cl2B—Sn1—Cl2 | 90.21 (15)     |
| Cl2B—Sn1—Cl2 | 89.79 (15)     |
| Cl2B—Sn1—Cl2 | 89.79 (15)     |
| Cl2B—Sn1—Cl1B | 90.21 (15)    |
| Cl2B—Sn1—Cl1B | 180.0          |
| Cl2B—Sn1—Cl3B | 87.92 (17)    |
| Cl2B—Sn1—Cl3B | 87.92 (17)     |
| Cl2B—Sn1—Cl3B | 92.08 (17)     |
| Cl2B—Sn1—Cl3B | 87.92 (17)     |
| Cl2B—Sn1—Cl3B | 92.08 (17)     |
| Cl2B—Sn1—Cl1A | 166.23 (14)    |
| Cl2B—Sn1—Cl1A | 13.77 (14)     |
| Cl2B—Sn1—Cl1A | 101.89 (14)    |

| Bond/Angle | Distance/Angle |
|------------|----------------|
| N1—C1—C2  | −6 (3)         |
| C1—N1—C5—C4 | 6 (2)        |
| N1—C1—C2—C3 | 2 (3)        |
| C1—C2—C3—C4 | 0 (3)        |

IUCrData (2022). 7, x220195
\[
\begin{array}{cccc}
C1—C2—C3—C6 & 178.1 (16) & C2—C3—C4—C5 & 0 (3) \\
C6—C3—C4—C5 & -177.7 (14) & C3—C4—C5—N1 & -3 (2) \\
\end{array}
\]

Symmetry code: (i) −x, −y+1, −z+1.

\textit{Hydrogen-bond geometry (Å, °)}

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|--------|
| N1—H1···O1W \text{A}^{ii} | 0.86 | 1.96 | 2.760 (15) | 154 |
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| N3—H3B···Cl1B \text{B}^{iv} | 0.86 | 2.56 | 3.420 (16) | 175 |
| O1WA—H1WA···Cl2\text{A} | 0.85 | 2.77 | 3.415 (8) | 134 |
| O1WA—H1WB···Cl3A \text{A}^{vi} | 0.85 | 2.41 | 3.154 (9) | 147 |
| O1WB—H1WC···Cl1A \text{A}^{v} | 0.85 | 2.60 | 3.305 (10) | 142 |
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| C1—H1A···Cl3A \text{A}^{vii} | 0.93 | 2.67 | 3.561 (17) | 161 |
| C1—H1A···Cl3B \text{A}^{vii} | 0.93 | 2.43 | 3.356 (17) | 174 |
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Symmetry codes: (i) −x, −y+1, −z+1; (ii) −x+1, −y+4, −z+2; (iii) x, y−1, z; (iv) −x+1, −y+2, −z+2; (v) x, y+1, z+1; (vi) x, y+2, z+1; (vii) −x+1, −y+3, −z+2; (viii) −x+1, −y+2, −z+1.