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ISSN 2414-3146

4-Amidinopyridinium hexachloridostannate(IV) dihydrate

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Received 12 January 2022
Accepted 18 February 2022

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

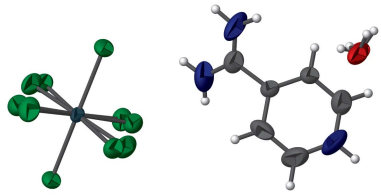
Keywords: crystal structure; hexachloridostannate(IV); 4-amidinopyridinium.

CCDC reference: 2153109

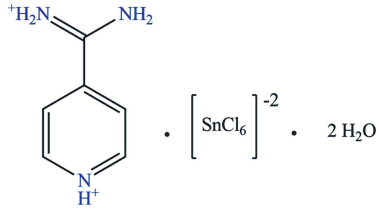
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrated molecular salt {systematic name: 4-[amino(iminiumyl)methyl]pyridin-1-ium hexachloridostannate(IV) dihydrate}, $(C_6H_9N_3)[SnCl_6]\cdot 2H_2O$, the tin atom lies on a crystallographic inversion centre and the organic cation shows whole-molecule disorder. Numerous N—H···O, N—H···Cl and O—H···Cl hydrogen bonds link the components in the crystal.

3D view



Chemical scheme



Structure description

The title hydrated molecular salt, with formula $(C_6H_9N_3)\cdot[SnCl_6]\cdot 2H_2O$, crystallizes in the triclinic space group $P\overline{1}$. The asymmetric unit is constituted by a $Sn_{0.5}Cl_3$ fragment (Sn site symmetry $\overline{1}$), 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 Cl3*A*/Cl3*B*), 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).

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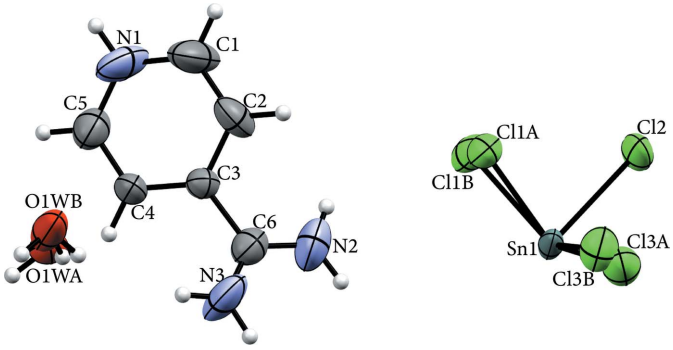

Figure 1
The molecular structure showing 30% displacement ellipsoids.

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.

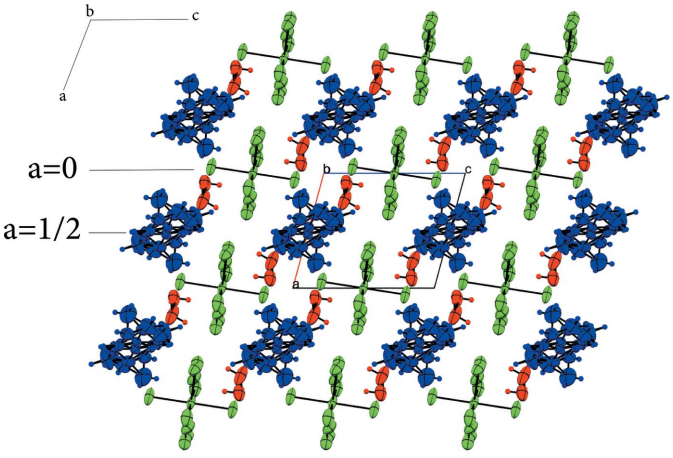

Figure 2
Projection of the crystal packing on the *ac* plane.

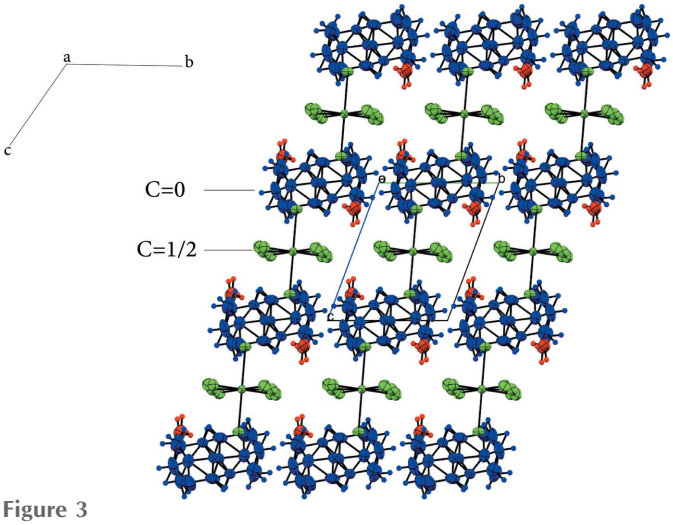

Figure 3
Projection of the crystal packing on the *bc* plane.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1WA^{i}	0.86	1.96	2.760 (15)	154
N1—H1···O1WB^{i}	0.86	1.87	2.649 (15)	149
N2—H2A···Cl2^{ii}	0.86	2.68	3.431 (11)	147
N3—H3A···O1WA^{iii}	0.86	2.13	2.961 (16)	162
N3—H3A···O1WB^{iii}	0.86	1.96	2.795 (16)	163
N3—H3B···Cl1A^{iv}	0.86	2.69	3.093 (16)	110
N3—H3B···Cl3B^{iv}	0.86	2.56	3.420 (16)	175
O1WA—H1WA···Cl2^{v}	0.85	2.77	3.415 (8)	134
O1WA—H1WB···Cl3A^{vi}	0.85	2.41	3.154 (9)	147
O1WB—H1WC···Cl1A^{v}	0.85	2.60	3.305 (10)	142
O1WB—H1WC···Cl1B^{v}	0.85	2.36	3.085 (10)	144
O1WB—H1WC···Cl1A^{vii}	0.85	2.69	3.251 (12)	124
O1WB—H1WC···Cl1B^{vii}	0.85	2.83	3.396 (13)	126
C1—H1A···Cl3A^{viii}	0.93	2.67	3.561 (17)	161
C1—H1A···Cl3B^{viii}	0.93	2.43	3.356 (17)	174
C5—H5···Cl1A^{vii}	0.93	2.80	3.674 (12)	157
C5—H5···Cl1B^{vii}	0.93	2.56	3.385 (12)	149

Symmetry codes: (i) $-x+1, -y+4, -z+2$; (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) $-x+1, -y+2, -z+1$.

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	
Chemical formula	$(C_6H_9N_3)[SnCl_6]\cdot 2H_2O$
M_r	490.58
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	7.4224 (13), 7.4518 (11), 8.4986 (16)
α, β, γ (°)	105.726 (7), 97.426 (9), 112.383 (7)
V (Å^3)	403.85 (12)
Z	1
Radiation type	Mo $K\alpha$
μ (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}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (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 Developpement Technologique DGRSDT Algérie.

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full crystallographic data

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

4-Amidinopyridinium hexachloridostannate(IV) dihydrate

Rochdi Ghallab, Hassiba Bougueria and Hocine Merazig

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

Crystal data

$(C_6H_9N_3)[SnCl_6]\cdot 2H_2O$
$M_r = 490.58$
Triclinic, $P\overline{1}$
$a = 7.4224$ (13) Å
$b = 7.4518$ (11) Å
$c = 8.4986$ (16) Å
$\alpha = 105.726$ (7)°
$\beta = 97.426$ (9)°
$\gamma = 112.383$ (7)°
$V = 403.85$ (12) Å^3
$Z = 1$
$F(000) = 238$
$D_x = 2.017$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1889 reflections
$\theta = 5.0$–$30.5°$
$\mu = 2.57$ mm^{-1}
$T = 296$ K
Block, colourless
0.17 × 0.13 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.676$, $T_{max} = 0.754$
10469 measured reflections
2442 independent reflections
1889 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 30.5°$, $\theta_{min} = 5.0°$
$h = -10\rightarrow10$
$k = -10\rightarrow10$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.085$
$S = 1.15$
2442 reflections
154 parameters
53 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0167P)^2 + 0.8036P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.22$ e Å^{-3}
$\Delta\rho_{min} = -1.35$ e Å^{-3}

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*	U_{iso}*/U_{eq}	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)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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, º)

Sn1—Cl2	2.4470 (10)	C3—C4	1.372 (15)
Sn1—Cl2^{i}	2.4470 (10)	C3—C2	1.366 (15)
Sn1—Cl1B^{i}	2.371 (6)	C3—C6	1.48 (2)
Sn1—Cl1B	2.371 (6)	C4—C5	1.354 (10)
Sn1—Cl3B^{i}	2.451 (7)	C5—N1	1.339 (11)
Sn1—Cl3B	2.451 (7)	N1—C1	1.358 (12)
Sn1—Cl1A	2.475 (7)	C1—C2	1.374 (12)
Sn1—Cl1A^{i}	2.475 (7)	C6—N2	1.306 (14)
Sn1—Cl3A^{i}	2.402 (4)	C6—N3	1.226 (16)
Sn1—Cl3A	2.402 (4)		
Cl2—Sn1—Cl2^{i}	180.0	Cl1B—Sn1—Cl3A^{i}	78.11 (14)
Cl2—Sn1—Cl3B^{i}	87.17 (16)	Cl3B—Sn1—Cl3B^{i}	180.0
Cl2^{i}—Sn1—Cl3B^{i}	92.83 (16)	Cl3B—Sn1—Cl1A^{i}	105.72 (16)
Cl2—Sn1—Cl3B	92.83 (16)	Cl3B^{i}—Sn1—Cl1A^{i}	74.28 (16)
Cl2^{i}—Sn1—Cl3B	87.17 (16)	Cl3A—Sn1—Cl2	89.55 (8)
Cl2^{i}—Sn1—Cl1A	90.98 (14)	Cl3A^{i}—Sn1—Cl2	90.45 (8)
Cl2—Sn1—Cl1A	89.02 (14)	Cl3A—Sn1—Cl1A	88.16 (12)
Cl2—Sn1—Cl1A^{i}	90.98 (14)	Cl3A^{i}—Sn1—Cl1A	91.84 (12)
Cl2^{i}—Sn1—Cl1A^{i}	89.02 (14)	Cl3A^{i}—Sn1—Cl3A	180.0
Cl1B—Sn1—Cl2^{i}	89.79 (15)	C4—C3—C6	123.3 (12)
Cl1B—Sn1—Cl2	90.21 (15)	C2—C3—C4	119.7 (15)
Cl1B^{i}—Sn1—Cl2	89.79 (15)	C2—C3—C6	117.0 (12)
Cl1B^{i}—Sn1—Cl2^{i}	90.21 (15)	C5—C4—C3	120.1 (10)
Cl1B^{i}—Sn1—Cl1B	180.0	N1—C5—C4	120.0 (9)
Cl1B—Sn1—Cl3B	87.92 (17)	C5—N1—C1	121.3 (9)
Cl1B^{i}—Sn1—Cl3B	92.08 (17)	N1—C1—C2	119.2 (10)
Cl1B^{i}—Sn1—Cl3B^{i}	87.92 (17)	C3—C2—C1	119.5 (11)
Cl1B—Sn1—Cl3B^{i}	92.08 (17)	N2—C6—C3	116.5 (11)
Cl1B—Sn1—Cl1A^{i}	166.23 (14)	N3—C6—C3	118.0 (11)
Cl1B^{i}—Sn1—Cl1A^{i}	13.77 (14)	N3—C6—N2	125.4 (11)
Cl1B^{i}—Sn1—Cl3A^{i}	101.89 (14)		
C5—N1—C1—C2	−6 (3)	C2—C3—C6—N2	−42 (2)
C1—N1—C5—C4	6 (2)	C2—C3—C6—N3	142.5 (16)
N1—C1—C2—C3	2 (3)	C4—C3—C6—N2	135.9 (17)
C1—C2—C3—C4	0 (3)	C4—C3—C6—N3	−40 (3)

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)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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O1*WB*—H1*WC*···Cl1*A*^{v}	0.85	2.60	3.305 (10)	142
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