

Bis(2-carboxyquinolinium) hexachloridostannate(IV) dihydrate

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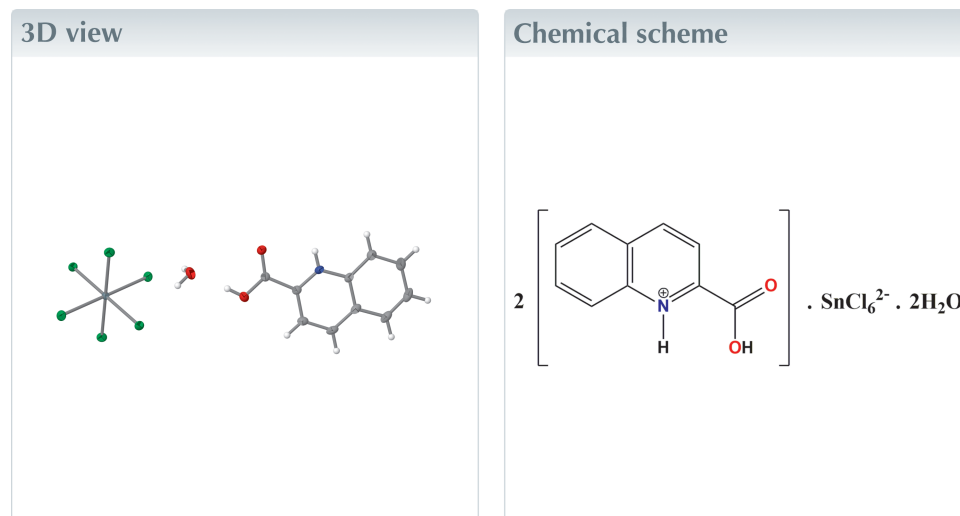
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Keywords: crystal structure; $[\text{SnCl}_6]^{2-}$ anion; hydrogen-bonding; π - π stacking.

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

In the hydrated title salt, $(\text{C}_{10}\text{H}_8\text{NO}_2)_2[\text{SnCl}_6]\cdot 2\text{H}_2\text{O}$, the tin(IV) atom is located about a center of inversion. In the crystal structure, the octahedral inorganic anion and the water molecule of crystallization interact through $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds, supplemented by weak π - π stacking between neighboring cations, and $\text{C}-\text{Cl}\cdots\pi$ interactions.



Structure description

The crystal structure determination of the title compound, (I), was undertaken as part of studies of organic–inorganic hybrid materials, which may exhibit various interesting physical properties such as dielectric characteristics (Hajlaoui *et al.*, 2013).

The asymmetric unit of (I) comprises half of an octahedral $[\text{SnCl}_6]^{2-}$ anion (the whole anion being completed by inversion symmetry), one 2-carboxyquinolinium cation, and a water molecule of crystallization (Fig. 1).

The Sn^{IV} atom is coordinated by six chlorine atoms, forming a slightly distorted octahedron. The lengths of the $\text{Sn}-\text{Cl}$ bonds in the hexachloridostannate(IV) anion range from 2.4180 (3) to 2.4406 (3) Å and the $\text{Cl}-\text{Sn}-\text{Cl}$ bond angles deviate only by approximately 1° from ideal values, similar to those reported in the literature (Ghallab *et al.*, 2020; Rademeyer, 2004; Billing *et al.*, 2007).

In the 2-carboxyquinolinium cation, the $\text{C}-\text{C}$ bond lengths range from 1.364 (2) to 1.5029 (15) Å and the $\text{C}-\text{N}$ bond lengths are 1.3282 (14) and 1.3649 (17) Å; the angles vary between 115.14 (10) ($\text{N1}-\text{C2}-\text{C1}$) and 126.71 (11) $^\circ$ ($\text{O1}-\text{C}-\text{O2}$). These values are similar compared with related cations with protonated aromatic N atoms (Gelmboldt *et al.*, 2007; Smith *et al.*, 2004, 2008). The cation in (I) is not planar, as indicated by a dihedral angle between the quinoline ring and the carboxy group of 11.61 (9) $^\circ$.

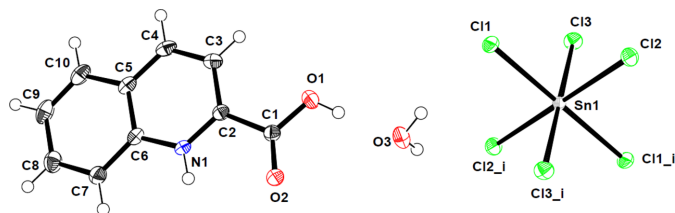


Figure 1

View of the molecular entities in (I) with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. [Symmetry code: (i) $-x, -y, -z$].

Apart from Coulombic forces, the cohesion in the crystal structure is ensured by classical hydrogen bonds between the carboxyl group as donor and the water molecule as acceptor groups, and by interactions between the protonated quinoline N atom and the non-protonated O atom of the carboxyl group of a neighboring molecule. Further interactions involve the water molecule and the hexachloridostannate anion (Table 1, Fig. 2). There are also π - π stacking interactions between neighboring cations [3.7898 (8) Å, slippage 1.678 Å; Fig. 3a] and Cl \cdots π interactions [3.5633 (6) Å; Fig. 3b].

Synthesis and crystallization

Tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) was mixed with quinaldic acid ($\text{C}_{10}\text{H}_7\text{NO}_2$) in a 1:2 molar ratio, along with a few drops of hydrochloric acid in distilled water. The mixture was refluxed for one h at 343 K. After two weeks of slow solvent evaporation at room temperature, colorless single crystals suitable for X-ray analysis were obtained.

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 2. The H atom bound to the N atom was refined freely. 14 reflections were omitted from the refinement because they were obstructed from the beam stop.

Acknowledgements

Thanks are due to DRSDT-Algeria.

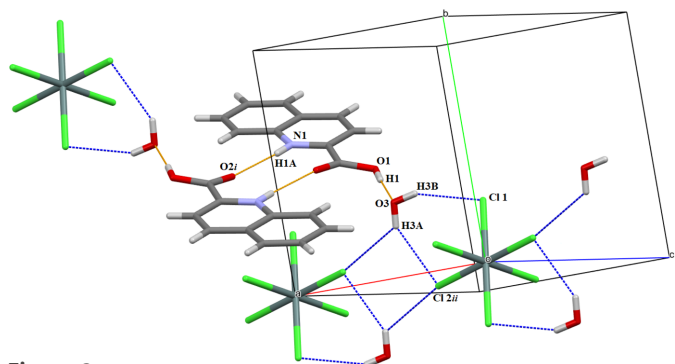


Figure 2

O-H \cdots O, N-H \cdots O and O-H \cdots Cl hydrogen-bonding interactions in the crystal structure of (I) indicated by dashed lines. Symmetry codes refer to Table 1.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1-H1 \cdots O3	0.82	1.75	2.5684 (14)	176
N1-H1A \cdots O2 ⁱ	0.832 (17)	2.028 (17)	2.8348 (13)	163.4 (15)
O3-H3A \cdots Cl1	0.85	2.46	3.2048 (11)	146
O3-H3B \cdots Cl2 ⁱⁱ	0.85	2.61	3.3708 (11)	150

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

Table 2

Experimental details.

Crystal data	
Chemical formula	$(\text{C}_{10}\text{H}_8\text{NO}_2)[\text{SnCl}_6] \cdot 2\text{H}_2\text{O}$
M_r	715.77
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	296
a, b, c (Å)	8.3220 (4), 9.2704 (4), 9.4248 (4)
α, β, γ (°)	108.101 (2), 99.515 (2), 99.749 (2)
V (Å ³)	662.46 (5)
Z	1
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	1.61
Crystal size (mm)	0.10 \times 0.09 \times 0.08
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
$T_{\text{min}}, T_{\text{max}}$	0.851, 0.879
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19467, 3262, 3199
R_{int}	0.015
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.013, 0.033, 1.08
No. of reflections	3262
No. of parameters	168
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.41, -0.25

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *OLEX2.solve* (Bourhis *et al.*, 2015), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

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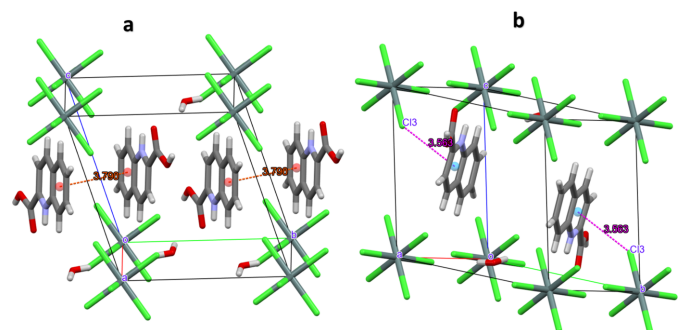


Figure 3

(a) π - π stacking interactions and (b) Cl \cdots π interactions in the crystal structure of (I).

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full crystallographic data

IUCrData (2024). **9**, x240826 [<https://doi.org/10.1107/S2414314624008265>]

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Bis(2-carboxyquinolinium) hexachloridostannate(IV) dihydrate

Crystal data

$(C_{10}H_8NO_2)[SnCl_6] \cdot 2H_2O$

$M_r = 715.77$

Triclinic, $P\bar{1}$

$a = 8.3220$ (4) Å

$b = 9.2704$ (4) Å

$c = 9.4248$ (4) Å

$\alpha = 108.101$ (2)°

$\beta = 99.515$ (2)°

$\gamma = 99.749$ (2)°

$V = 662.46$ (5) Å³

$Z = 1$

$F(000) = 354$

$D_x = 1.794$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9611 reflections

$\theta = 3.7$ – 48.2 °

$\mu = 1.61$ mm⁻¹

$T = 296$ K

Plate, yellow

$0.10 \times 0.09 \times 0.08$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.851$, $T_{\max} = 0.879$

19467 measured reflections

3262 independent reflections

3199 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 4.8$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.013$

$wR(F^2) = 0.033$

$S = 1.08$

3262 reflections

168 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.013P)^2 + 0.3169P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.41$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.000000	0.000000	0.000000	0.01307 (3)
Cl1	0.12549 (3)	0.26567 (3)	0.17700 (3)	0.01985 (6)
Cl2	-0.27553 (3)	0.05974 (3)	-0.01450 (3)	0.02261 (6)
Cl3	-0.03577 (4)	-0.08014 (3)	0.21592 (3)	0.02122 (6)
O2	0.82893 (11)	0.48826 (10)	0.04894 (9)	0.02362 (17)
O1	0.70770 (11)	0.47285 (12)	0.24138 (10)	0.02734 (19)
H1	0.625567	0.421382	0.171718	0.041*
O3	0.44565 (12)	0.30407 (12)	0.03220 (12)	0.0316 (2)
H3A	0.358189	0.328438	0.058776	0.047*
H3B	0.438485	0.207555	0.016123	0.047*
N1	1.12611 (12)	0.62982 (11)	0.25392 (11)	0.01629 (17)
C2	0.98812 (14)	0.60663 (12)	0.30529 (12)	0.0171 (2)
C1	0.83151 (14)	0.51554 (13)	0.18355 (13)	0.0183 (2)
C3	0.99422 (16)	0.66408 (14)	0.46178 (13)	0.0213 (2)
H3	0.897184	0.649793	0.497561	0.026*
C6	1.28072 (14)	0.70531 (13)	0.34788 (13)	0.0181 (2)
C9	1.59120 (17)	0.85434 (15)	0.54706 (16)	0.0299 (3)
H9	1.696466	0.902964	0.612341	0.036*
C8	1.57523 (16)	0.79829 (15)	0.38631 (16)	0.0277 (3)
H8	1.670263	0.812255	0.347661	0.033*
C7	1.42258 (15)	0.72382 (14)	0.28635 (14)	0.0230 (2)
H7	1.413118	0.686630	0.180899	0.028*
C5	1.29308 (15)	0.76284 (13)	0.50879 (13)	0.0208 (2)
C10	1.45388 (17)	0.83779 (15)	0.60721 (14)	0.0274 (3)
H10	1.465775	0.875747	0.712974	0.033*
C4	1.14626 (16)	0.74234 (14)	0.56255 (13)	0.0230 (2)
H4	1.151849	0.782030	0.667260	0.028*
H1A	1.119 (2)	0.5947 (19)	0.160 (2)	0.026 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01189 (5)	0.01279 (5)	0.01377 (5)	0.00232 (3)	0.00262 (3)	0.00407 (4)
Cl1	0.02182 (13)	0.01429 (11)	0.01922 (12)	0.00029 (9)	0.00555 (10)	0.00173 (9)
Cl2	0.01509 (12)	0.02717 (14)	0.02324 (13)	0.00878 (10)	0.00250 (10)	0.00457 (11)
Cl3	0.02591 (13)	0.02106 (13)	0.01886 (12)	0.00433 (10)	0.00618 (10)	0.01006 (10)
O2	0.0205 (4)	0.0291 (4)	0.0164 (4)	0.0005 (3)	0.0033 (3)	0.0045 (3)
O1	0.0201 (4)	0.0358 (5)	0.0226 (4)	-0.0002 (4)	0.0071 (3)	0.0077 (4)
O3	0.0207 (4)	0.0325 (5)	0.0368 (5)	-0.0007 (4)	0.0106 (4)	0.0069 (4)
N1	0.0185 (4)	0.0158 (4)	0.0123 (4)	0.0036 (3)	0.0020 (3)	0.0028 (3)
C2	0.0197 (5)	0.0152 (5)	0.0164 (5)	0.0049 (4)	0.0035 (4)	0.0053 (4)
C1	0.0182 (5)	0.0175 (5)	0.0187 (5)	0.0047 (4)	0.0046 (4)	0.0051 (4)
C3	0.0257 (6)	0.0217 (5)	0.0175 (5)	0.0074 (4)	0.0075 (4)	0.0063 (4)
C6	0.0196 (5)	0.0150 (5)	0.0167 (5)	0.0038 (4)	0.0005 (4)	0.0037 (4)
C9	0.0239 (6)	0.0260 (6)	0.0301 (6)	0.0012 (5)	-0.0089 (5)	0.0069 (5)

C8	0.0201 (6)	0.0267 (6)	0.0330 (7)	0.0044 (5)	0.0023 (5)	0.0084 (5)
C7	0.0210 (6)	0.0235 (6)	0.0213 (5)	0.0036 (4)	0.0029 (4)	0.0051 (5)
C5	0.0258 (6)	0.0164 (5)	0.0164 (5)	0.0044 (4)	-0.0003 (4)	0.0037 (4)
C10	0.0310 (7)	0.0246 (6)	0.0188 (5)	0.0035 (5)	-0.0053 (5)	0.0042 (5)
C4	0.0326 (6)	0.0216 (5)	0.0132 (5)	0.0078 (5)	0.0035 (4)	0.0039 (4)

Geometric parameters (Å, °)

Sn1—Cl3	2.4180 (3)	C2—C1	1.5029 (15)
Sn1—Cl3 ⁱ	2.4180 (3)	C3—C4	1.3758 (17)
Sn1—Cl1	2.4370 (3)	C3—H3	0.9300
Sn1—Cl1 ⁱ	2.4370 (3)	C6—C7	1.4067 (17)
Sn1—Cl2 ⁱ	2.4406 (3)	C6—C5	1.4220 (15)
Sn1—Cl2	2.4406 (3)	C9—C10	1.364 (2)
O2—C1	1.2099 (14)	C9—C8	1.4155 (19)
O1—C1	1.3026 (14)	C9—H9	0.9300
O1—H1	0.8200	C8—C7	1.3715 (17)
O3—H3A	0.8511	C8—H8	0.9300
O3—H3B	0.8496	C7—H7	0.9300
N1—C2	1.3282 (14)	C5—C4	1.4041 (18)
N1—C6	1.3649 (14)	C5—C10	1.4171 (17)
N1—H1A	0.832 (17)	C10—H10	0.9300
C2—C3	1.3927 (15)	C4—H4	0.9300
Cl3—Sn1—Cl3 ⁱ	179.999 (12)	O1—C1—C2	112.24 (10)
Cl3—Sn1—Cl1	89.336 (10)	C4—C3—C2	118.86 (11)
Cl3 ⁱ —Sn1—Cl1	90.665 (10)	C4—C3—H3	120.6
Cl3—Sn1—Cl1 ⁱ	90.663 (10)	C2—C3—H3	120.6
Cl3 ⁱ —Sn1—Cl1 ⁱ	89.336 (10)	N1—C6—C7	120.61 (10)
Cl1—Sn1—Cl1 ⁱ	180.0	N1—C6—C5	117.83 (10)
Cl3—Sn1—Cl2 ⁱ	91.121 (10)	C7—C6—C5	121.56 (11)
Cl3 ⁱ —Sn1—Cl2 ⁱ	88.879 (10)	C10—C9—C8	120.70 (12)
Cl1—Sn1—Cl2 ⁱ	90.704 (10)	C10—C9—H9	119.6
Cl1 ⁱ —Sn1—Cl2 ⁱ	89.295 (10)	C8—C9—H9	119.6
Cl3—Sn1—Cl2	88.879 (10)	C7—C8—C9	121.41 (12)
Cl3 ⁱ —Sn1—Cl2	91.121 (10)	C7—C8—H8	119.3
Cl1—Sn1—Cl2	89.295 (10)	C9—C8—H8	119.3
Cl1 ⁱ —Sn1—Cl2	90.706 (10)	C8—C7—C6	118.11 (11)
Cl2 ⁱ —Sn1—Cl2	180.0	C8—C7—H7	120.9
C1—O1—H1	109.5	C6—C7—H7	120.9
H3A—O3—H3B	109.4	C4—C5—C10	123.20 (11)
C2—N1—C6	123.38 (10)	C4—C5—C6	118.68 (11)
C2—N1—H1A	118.5 (11)	C10—C5—C6	118.11 (11)
C6—N1—H1A	118.2 (11)	C9—C10—C5	120.10 (12)
N1—C2—C3	120.58 (10)	C9—C10—H10	120.0
N1—C2—C1	115.14 (10)	C5—C10—H10	120.0
C3—C2—C1	124.29 (10)	C3—C4—C5	120.63 (11)
O2—C1—O1	126.71 (11)	C3—C4—H4	119.7

O2—C1—C2	121.05 (10)	C5—C4—H4	119.7
C6—N1—C2—C3	1.99 (16)	N1—C6—C7—C8	-178.90 (11)
C6—N1—C2—C1	-177.71 (10)	C5—C6—C7—C8	0.58 (18)
N1—C2—C1—O2	-11.11 (16)	N1—C6—C5—C4	-1.35 (16)
C3—C2—C1—O2	169.20 (11)	C7—C6—C5—C4	179.15 (11)
N1—C2—C1—O1	168.63 (10)	N1—C6—C5—C10	178.41 (10)
C3—C2—C1—O1	-11.06 (16)	C7—C6—C5—C10	-1.09 (17)
N1—C2—C3—C4	-1.40 (17)	C8—C9—C10—C5	0.5 (2)
C1—C2—C3—C4	178.27 (11)	C4—C5—C10—C9	-179.70 (12)
C2—N1—C6—C7	178.92 (11)	C6—C5—C10—C9	0.55 (18)
C2—N1—C6—C5	-0.58 (16)	C2—C3—C4—C5	-0.54 (18)
C10—C9—C8—C7	-1.0 (2)	C10—C5—C4—C3	-177.86 (12)
C9—C8—C7—C6	0.47 (19)	C6—C5—C4—C3	1.89 (17)

Symmetry code: (i) $-x, -y, -z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O3	0.82	1.75	2.5684 (14)	176
N1—H1A \cdots O2 ⁱⁱ	0.832 (17)	2.028 (17)	2.8348 (13)	163.4 (15)
O3—H3A \cdots C11	0.85	2.46	3.2048 (11)	146
O3—H3B \cdots C12 ⁱ	0.85	2.61	3.3708 (11)	150
C3—H3 \cdots C11 ⁱⁱⁱ	0.93	2.97	3.6014 (12)	127
C7—H7 \cdots O2 ⁱⁱ	0.93	2.58	3.2850 (15)	133
C7—H7 \cdots O3 ⁱⁱ	0.93	2.51	3.3144 (16)	146

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