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2-(2-Ammoniophenylamino)ethanaminium hexachlorostannate(IV) monohydrate

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Key indicators

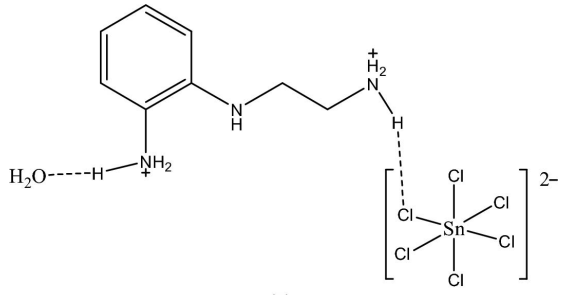
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.034
wR factor = 0.071
Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $(C_8H_{15}N_3)[SnCl_6]\cdot H_2O$, is a salt involving a doubly protonated amine cation and a divalent $[SnCl_6]^{2-}$ anion. The solid-state structure agrees with that found by ^{1}H NMR and ^{13}C NMR analyses. The salt has potential for use in the synthesis of organometallic compounds.

Comment

The title salt, (I), has found use in fields as varied as medical and organic intermediates. Furthermore, many metal complexes of ethylenediamine and its derivatives are biologically active. They can resist cancers and viruses, and can inhibit bacterial growth. Thus, in order to learn more about these compounds, we have synthesized compound (I), and report its structure here.



(I)

Compound (I) (Fig. 1) has been characterized by ^{1}H and ^{13}C NMR analyses and contains a doubly protonated amine cation and an $[SnCl_6]^{2-}$ anion. The amine cation is essentially planar (±0.028 Å), with the water molecule and the $[SnCl_6]^{2-}$ anion located at opposite ends of the cation. There are two hydrogen bonds, one between N1 and the water molecule [N1—H1c = 0.89 Å, H1c···O1 = 1.85 Å, N1···O1 = 2.736 (6) Å and N1—H1c···O1 = 171] and a second between N3 and a Cl atom of

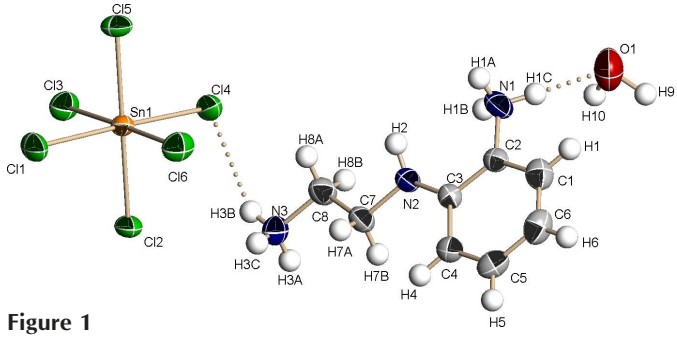


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

the anion [N3—H3*B* = 0.89 Å, H3*B*···Cl4 = 2.64 Å, N3···Cl4 = 3.397 (10) Å and N3—H3*B*···Cl4 = 143]. The $[SnCl_6]^{2-}$ anion has a distorted octahedral coordination, with the nearest Sn···N distance being Sn···N3 of 4.317 (5) Å.

Experimental

The title salt was synthesized according to Allali *et al.* (2004). 2-Nitrochlorobenzene and excess ethylenediamine were stirred for 1 h under reflux. After the excess of ethylenediamine had been distilled off, the crude extract was acidified at pH 6 with 2 N HCl, then heated and filtered. After cooling, *N*-(2-nitrophenyl)ethylenediamine hydrochloride was collected as yellow needles. The yellow needles were then reduced with $SnCl_2$ in aqueous HCl until there was no color visible. This solution was stewed for about 10 d, after which orange crystals suitable for data collection were obtained. ^{1}H NMR ($CDCl_3$): δ H_1 (7.27, *d*, 1H), H_4 (6.86, *d*, 1H), H_5 (7.13, *d*, 1H), H_6 (6.89, *d*, 1H), H_7 (3.41, *s*, 2H), H_8 (3.11, *s*, 2H), ^{13}C NMR ($CDCl_3$): δ C_1 (137.46), C_2 (127.57), C_3 (111.56), C_4 (121.29), C_5 (116.69), C_6 (115.01), C_7 (37.44), C_8 (35.41).

Crystal data

$(C_8H_{15}N_3)[SnCl_6]\cdot H_2O$
M_r = 502.64
Monoclinic, $P2_1/n$
a = 7.2640 (17) Å
b = 22.960 (6) Å
c = 10.270 (2) Å
β = 92.985 (3)°
V = 1710.6 (7) Å^3
Z = 4
D_x = 1.952 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3683 reflections
θ = 3.0–26.8°
μ = 2.42 mm^{-1}
T = 293 (2) K
Block, orange
0.3 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.530, T_{max} = 0.643
6951 measured reflections
3011 independent reflections
2653 reflections with $I > 2\sigma a(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = -8 \rightarrow 7$
$k = -27 \rightarrow 13$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.071
S = 1.07
3011 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.2954P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—Cl5	2.4046 (11)	Sn1—Cl2	2.4418 (11)
Sn1—Cl6	2.4082 (13)	O1—H10	1.041 (10)
Sn1—Cl4	2.4247 (11)	O1—H9	1.046 (10)
Sn1—Cl1	2.4343 (11)	N2—H2	0.862 (10)
Sn1—Cl3	2.4398 (13)		
Cl5—Sn1—Cl6	90.03 (4)	Cl4—Sn1—Cl3	90.25 (4)
Cl5—Sn1—Cl4	91.54 (4)	Cl1—Sn1—Cl3	88.61 (4)
Cl6—Sn1—Cl4	90.95 (4)	Cl5—Sn1—Cl2	179.34 (4)
Cl5—Sn1—Cl1	90.64 (4)	Cl6—Sn1—Cl2	90.49 (4)
Cl6—Sn1—Cl1	90.15 (4)	Cl4—Sn1—Cl2	88.04 (4)
Cl4—Sn1—Cl1	177.55 (4)	Cl1—Sn1—Cl2	89.77 (4)
Cl5—Sn1—Cl3	90.74 (4)	Cl3—Sn1—Cl2	88.75 (4)
Cl6—Sn1—Cl3	178.55 (4)	C3—N2—C7	120.8 (3)
C7—N2—C3—C2	176.1 (4)	N1—C2—C3—N2	−2.1 (6)
C7—N2—C3—C4	−3.0 (6)	N2—C3—C4—C5	179.1 (4)
C1—C2—C3—N2	−179.1 (4)	N2—C7—C8—N3	179.3 (4)

H2 attached to N2 and H9, H10 attached to O1 were located in a difference map and refined with distance restraints, N---H = 0.86 (1) Å and O---H = 1.05 (1) Å. Other H atoms were positioned geometrically and refined as riding, with C---H = 0.93 or 0.97, N---H = 0.89 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(N).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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