Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Triethylammonium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

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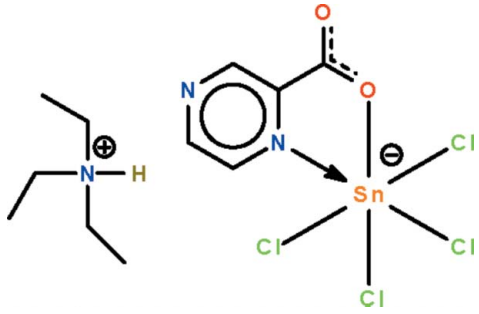
Received 26 April 2011; accepted 2 May 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.022; wR factor = 0.052; data-to-parameter ratio = 21.1.

The Sn^{IV} atom in the title ammonium stannate, $(Et_3NH)[Sn(C_5H_3N_2O_2)Cl_4]$, is chelated by an pyrazine-2-carboxylate ligand and exists in a *cis*-$SnCl_4NO$ octahedral geometry. The cation and the anion are linked by an N—H···N hydrogen bond.

Related literature

For triethylammonium tetrachlorido(pyridine-2-carboxylato)stannate(IV), see: Najafi *et al.* (2011).



Experimental

Crystal data

$(C_6H_{16}N)[Sn(C_5H_3N_2O_2)Cl_4]$
M_r = 485.78
Triclinic, $P\overline{1}$
a = 7.4497 (2) Å
b = 9.9752 (3) Å
c = 12.3728 (4) Å
α = 86.491 (2)°
β = 80.125 (3)°
γ = 83.817 (2)°
V = 899.70 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.02 mm^{-1}
T = 100 K
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.538, T_{max} = 0.632
15321 measured reflections
4094 independent reflections
3800 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.052
S = 1.02
4094 reflections
194 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1···N2	0.91 (3)	2.10 (3)	2.999 (2)	167 (2)

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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, 2010).

We thank Shahid Beheshti University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QK2007).

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Najafi, E., Amini, M. M. & Ng, S. W. (2011). *Acta Cryst.* E**67**, m351.
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supporting information

Acta Cryst. (2011). E**67**, m711 [doi:10.1107/S1600536811016473]

Triethylammonium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

S1. Comment

We have recently reported the crystal structure of triethylammonium tetrachlorido(pyridine-2-carboxylato)stannate, which was synthesized by the reaction of triethylammonium pyridine-2-carboxylate and stannic chloride. The Sn^{IV} atom in the anion is *N*,*O*-chelated by the pyridine-2-carboxylate in a *cis*-$SnCl_4NO$ octahedral geometry (Najafi *et al.*, 2011). In our previous studies, we have reacted aromatic carboxylic acid with stannic chloride, with/without a proton-abstraction agent. In the present study, replacing pyrdine-2-carboxylic acid by pyrazine-2-carboxylic acid affords a similar salt, $(Et_3NH)^+$ $[SnCl_4(C_5H_3N_2O_2)]^-$ (Scheme I, Fig. 1). The tin atom in the stannate is chelated by the pyrazine-2-carboxylate group in a *cis*-$SnCl_4NO$ octahedral geometry. The cation forms an N–H···N hydrogen bond with the anion. Of the four Sn–Cl bonds, the ones that are *trans* to the Sn–O/Sn–N bonds are somewhat shorter than the other two. No Cl···Cl interactions are present.

S2. Experimental

The reaction was carried out under a nitrogen atmosphere. Pyrazine-2-carboxylic acid (1.0 mmol, 0.12 g) and the triethylamine (1.0 mmol, 0.10 g) were dissolved in dry methanol (20 ml). Stannic chloride (1.0 mmol, 0.35 g) was added to the mixture and stirred for 12 h. Suitable crystals were obtained by slow evaporation of the solvent.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C). The ammonium H-atom was located in a difference Fourier map, and was freely refined.

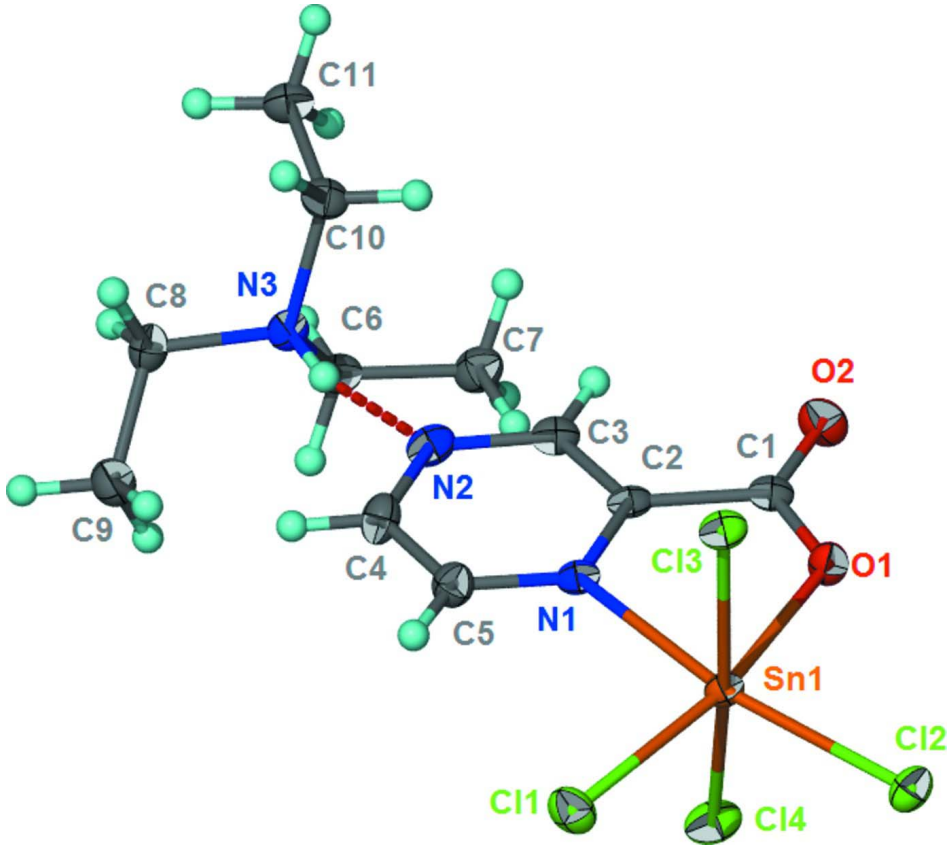

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $(Et_3NH)^+$ $[SnCl_4(C_5H_3N_2O_2)]$- at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The hydrogen bond is denoted by a dashed bond.

Triethylammonium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

Crystal data

$(C_6H_{16}N)[Sn(C_5H_3N_2O_2)Cl_4]$
$M_r = 485.78$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.4497$ (2) Å
$b = 9.9752$ (3) Å
$c = 12.3728$ (4) Å
$\alpha = 86.491$ (2)°
$\beta = 80.125$ (3)°
$\gamma = 83.817$ (2)°
$V = 899.70$ (5) Å^3
$Z = 2$
$F(000) = 480$
$D_x = 1.793$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10335 reflections
$\theta = 2.6$–29.2°
$\mu = 2.02$ mm^{-1}
$T = 100$ K
Irregular block, colorless
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.538$, $T_{max} = 0.632$
15321 measured reflections
4094 independent reflections
3800 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$

θ_{max} = 27.5°, θ_{min} = 2.6°
h = −9→9
k = −12→12
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.052
S = 1.02
4094 reflections
194 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 0.2896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.310753 (18)	0.248889 (13)	0.740250 (10)	0.01198 (5)
Cl1	0.46068 (7)	0.34171 (5)	0.86858 (4)	0.01988 (11)
Cl2	0.00680 (7)	0.26586 (5)	0.83792 (4)	0.01703 (11)
Cl3	0.39067 (7)	0.02423 (5)	0.80641 (4)	0.01605 (11)
Cl4	0.26028 (7)	0.46373 (5)	0.64356 (4)	0.02064 (11)
O1	0.22917 (19)	0.16094 (14)	0.60971 (11)	0.0149 (3)
O2	0.3047 (2)	0.10363 (15)	0.43513 (11)	0.0194 (3)
N1	0.5661 (2)	0.22659 (16)	0.61249 (13)	0.0128 (3)
N2	0.8334 (2)	0.21721 (17)	0.42643 (14)	0.0171 (4)
N3	1.0881 (2)	0.24246 (17)	0.21208 (14)	0.0142 (3)
C1	0.3419 (3)	0.14663 (19)	0.51771 (16)	0.0134 (4)
C2	0.5317 (3)	0.18609 (19)	0.51747 (16)	0.0128 (4)
C3	0.6667 (3)	0.18070 (19)	0.42525 (16)	0.0155 (4)
H3A	0.6397	0.1501	0.3592	0.019*
C4	0.8662 (3)	0.2544 (2)	0.52239 (17)	0.0179 (4)
H4A	0.9846	0.2789	0.5264	0.021*
C5	0.7345 (3)	0.2587 (2)	0.61660 (17)	0.0168 (4)
H5A	0.7638	0.2844	0.6837	0.020*
C6	0.9980 (3)	0.3321 (2)	0.13009 (16)	0.0161 (4)
H6A	1.0734	0.3221	0.0564	0.019*
H6B	0.9943	0.4272	0.1495	0.019*
C7	0.8062 (3)	0.3011 (2)	0.12490 (18)	0.0201 (5)
H7A	0.7565	0.3610	0.0691	0.030*
H7B	0.7288	0.3151	0.1966	0.030*
H7C	0.8084	0.2070	0.1057	0.030*
C8	1.2722 (3)	0.2874 (2)	0.22036 (17)	0.0173 (4)
H8A	1.3358	0.3118	0.1459	0.021*
H8B	1.3477	0.2116	0.2511	0.021*
C9	1.2544 (3)	0.4075 (2)	0.29232 (19)	0.0220 (5)
H9A	1.3763	0.4344	0.2947	0.033*
H9B	1.1952	0.3827	0.3668	0.033*

H9C	1.1801	0.4829	0.2620	0.033*
C10	1.1019 (3)	0.0937 (2)	0.19176 (16)	0.0168 (4)
H10A	1.1673	0.0429	0.2470	0.020*
H10B	0.9769	0.0646	0.2021	0.020*
C11	1.2003 (3)	0.0582 (2)	0.07822 (17)	0.0189 (4)
H11A	1.2039	−0.0392	0.0701	0.028*
H11B	1.3255	0.0840	0.0681	0.028*
H11C	1.1352	0.1067	0.0230	0.028*
H1	1.015 (3)	0.249 (2)	0.279 (2)	0.027 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01291 (8)	0.01297 (8)	0.00947 (8)	0.00012 (5)	−0.00092 (5)	−0.00091 (5)
Cl1	0.0232 (3)	0.0225 (3)	0.0154 (2)	−0.0057 (2)	−0.0037 (2)	−0.0045 (2)
Cl2	0.0141 (2)	0.0218 (3)	0.0137 (2)	0.00087 (19)	0.00009 (19)	−0.00038 (19)
Cl3	0.0189 (3)	0.0141 (2)	0.0142 (2)	0.00126 (19)	−0.00218 (19)	0.00034 (18)
Cl4	0.0249 (3)	0.0158 (2)	0.0181 (2)	0.0031 (2)	0.0002 (2)	0.00323 (19)
O1	0.0143 (7)	0.0201 (7)	0.0107 (7)	−0.0020 (6)	−0.0023 (6)	−0.0023 (6)
O2	0.0241 (8)	0.0229 (8)	0.0123 (7)	−0.0054 (6)	−0.0030 (6)	−0.0027 (6)
N1	0.0154 (9)	0.0120 (8)	0.0104 (8)	0.0013 (7)	−0.0017 (7)	−0.0008 (6)
N2	0.0182 (9)	0.0166 (9)	0.0150 (9)	0.0021 (7)	−0.0011 (7)	0.0008 (7)
N3	0.0151 (9)	0.0164 (9)	0.0103 (8)	−0.0020 (7)	0.0002 (7)	−0.0004 (7)
C1	0.0183 (10)	0.0092 (9)	0.0121 (9)	0.0000 (8)	−0.0028 (8)	0.0015 (7)
C2	0.0167 (10)	0.0088 (9)	0.0116 (9)	0.0016 (7)	−0.0011 (8)	0.0006 (7)
C3	0.0201 (11)	0.0130 (10)	0.0126 (10)	0.0001 (8)	−0.0023 (8)	0.0004 (8)
C4	0.0133 (10)	0.0222 (11)	0.0180 (10)	−0.0011 (8)	−0.0038 (8)	0.0028 (8)
C5	0.0157 (10)	0.0189 (10)	0.0161 (10)	−0.0010 (8)	−0.0040 (8)	−0.0005 (8)
C6	0.0195 (11)	0.0144 (10)	0.0139 (10)	0.0010 (8)	−0.0035 (8)	0.0002 (8)
C7	0.0199 (11)	0.0211 (11)	0.0190 (10)	0.0015 (9)	−0.0046 (9)	−0.0012 (9)
C8	0.0140 (10)	0.0213 (11)	0.0164 (10)	−0.0028 (8)	−0.0017 (8)	0.0005 (8)
C9	0.0199 (11)	0.0191 (11)	0.0283 (12)	−0.0030 (9)	−0.0062 (10)	−0.0019 (9)
C10	0.0207 (11)	0.0135 (10)	0.0158 (10)	−0.0022 (8)	−0.0022 (9)	0.0009 (8)
C11	0.0215 (11)	0.0176 (11)	0.0169 (10)	0.0008 (9)	−0.0018 (9)	−0.0031 (8)

Geometric parameters (Å, º)

Sn1—O1	2.0911 (13)	C4—H4A	0.9500
Sn1—N1	2.2558 (16)	C5—H5A	0.9500
Sn1—Cl2	2.3707 (5)	C6—C7	1.507 (3)
Sn1—Cl1	2.3742 (5)	C6—H6A	0.9900
Sn1—Cl3	2.3879 (5)	C6—H6B	0.9900
Sn1—Cl4	2.4150 (5)	C7—H7A	0.9800
O1—C1	1.299 (2)	C7—H7B	0.9800
O2—C1	1.217 (2)	C7—H7C	0.9800
N1—C5	1.337 (3)	C8—C9	1.517 (3)
N1—C2	1.340 (2)	C8—H8A	0.9900
N2—C3	1.334 (3)	C8—H8B	0.9900

N2—C4	1.334 (3)	C9—H9A	0.9800
N3—C6	1.508 (3)	C9—H9B	0.9800
N3—C8	1.509 (3)	C9—H9C	0.9800
N3—C10	1.510 (3)	C10—C11	1.513 (3)
N3—H1	0.91 (3)	C10—H10A	0.9900
C1—C2	1.508 (3)	C10—H10B	0.9900
C2—C3	1.385 (3)	C11—H11A	0.9800
C3—H3A	0.9500	C11—H11B	0.9800
C4—C5	1.389 (3)	C11—H11C	0.9800

O1—Sn1—N1	75.62 (6)	N1—C5—H5A	120.1
O1—Sn1—Cl2	91.17 (4)	C4—C5—H5A	120.1
N1—Sn1—Cl2	166.13 (4)	C7—C6—N3	113.48 (17)
O1—Sn1—Cl1	168.99 (4)	C7—C6—H6A	108.9
N1—Sn1—Cl1	93.47 (4)	N3—C6—H6A	108.9
Cl2—Sn1—Cl1	99.813 (18)	C7—C6—H6B	108.9
O1—Sn1—Cl3	86.56 (4)	N3—C6—H6B	108.9
N1—Sn1—Cl3	88.14 (4)	H6A—C6—H6B	107.7
Cl2—Sn1—Cl3	95.397 (18)	C6—C7—H7A	109.5
Cl1—Sn1—Cl3	91.748 (18)	C6—C7—H7B	109.5
O1—Sn1—Cl4	87.08 (4)	H7A—C7—H7B	109.5
N1—Sn1—Cl4	82.96 (4)	C6—C7—H7C	109.5
Cl2—Sn1—Cl4	92.285 (18)	H7A—C7—H7C	109.5
Cl1—Sn1—Cl4	93.058 (19)	H7B—C7—H7C	109.5
Cl3—Sn1—Cl4	170.120 (17)	N3—C8—C9	111.95 (17)
C1—O1—Sn1	119.66 (12)	N3—C8—H8A	109.2
C5—N1—C2	118.45 (17)	C9—C8—H8A	109.2
C5—N1—Sn1	129.40 (13)	N3—C8—H8B	109.2
C2—N1—Sn1	111.85 (13)	C9—C8—H8B	109.2
C3—N2—C4	116.56 (18)	H8A—C8—H8B	107.9
C6—N3—C8	110.51 (15)	C8—C9—H9A	109.5
C6—N3—C10	114.20 (15)	C8—C9—H9B	109.5
C8—N3—C10	111.59 (16)	H9A—C9—H9B	109.5
C6—N3—H1	108.5 (15)	C8—C9—H9C	109.5
C8—N3—H1	108.8 (15)	H9A—C9—H9C	109.5
C10—N3—H1	102.8 (15)	H9B—C9—H9C	109.5
O2—C1—O1	124.81 (19)	N3—C10—C11	113.47 (16)
O2—C1—C2	119.63 (17)	N3—C10—H10A	108.9
O1—C1—C2	115.56 (16)	C11—C10—H10A	108.9
N1—C2—C3	120.55 (18)	N3—C10—H10B	108.9
N1—C2—C1	116.58 (17)	C11—C10—H10B	108.9
C3—C2—C1	122.87 (17)	H10A—C10—H10B	107.7
N2—C3—C2	121.97 (18)	C10—C11—H11A	109.5
N2—C3—H3A	119.0	C10—C11—H11B	109.5
C2—C3—H3A	119.0	H11A—C11—H11B	109.5
N2—C4—C5	122.67 (19)	C10—C11—H11C	109.5
N2—C4—H4A	118.7	H11A—C11—H11C	109.5
C5—C4—H4A	118.7	H11B—C11—H11C	109.5

N1—C5—C4	119.72 (18)		
N1—Sn1—O1—C1	−6.70 (13)	C5—N1—C2—C1	178.09 (16)
Cl2—Sn1—O1—C1	169.01 (13)	Sn1—N1—C2—C1	−7.6 (2)
Cl1—Sn1—O1—C1	−14.2 (3)	O2—C1—C2—N1	−176.95 (18)
Cl3—Sn1—O1—C1	−95.65 (13)	O1—C1—C2—N1	2.4 (3)
Cl4—Sn1—O1—C1	76.78 (13)	O2—C1—C2—C3	2.6 (3)
O1—Sn1—N1—C5	−178.97 (18)	O1—C1—C2—C3	−178.07 (18)
Cl2—Sn1—N1—C5	162.83 (14)	C4—N2—C3—C2	2.5 (3)
Cl1—Sn1—N1—C5	−0.41 (17)	N1—C2—C3—N2	−1.1 (3)
Cl3—Sn1—N1—C5	−92.05 (17)	C1—C2—C3—N2	179.39 (18)
Cl4—Sn1—N1—C5	92.25 (17)	C3—N2—C4—C5	−1.5 (3)
O1—Sn1—N1—C2	7.47 (12)	C2—N1—C5—C4	2.4 (3)
Cl2—Sn1—N1—C2	−10.7 (3)	Sn1—N1—C5—C4	−170.79 (14)
Cl1—Sn1—N1—C2	−173.97 (12)	N2—C4—C5—N1	−1.0 (3)
Cl3—Sn1—N1—C2	94.39 (13)	C8—N3—C6—C7	−174.78 (16)
Cl4—Sn1—N1—C2	−81.31 (12)	C10—N3—C6—C7	58.4 (2)
Sn1—O1—C1—O2	−175.82 (15)	C6—N3—C8—C9	79.8 (2)
Sn1—O1—C1—C2	4.8 (2)	C10—N3—C8—C9	−151.96 (17)
C5—N1—C2—C3	−1.4 (3)	C6—N3—C10—C11	56.1 (2)
Sn1—N1—C2—C3	172.92 (15)	C8—N3—C10—C11	−70.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H1···N2	0.91 (3)	2.10 (3)	2.999 (2)	167 (2)