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3-Methoxycarbonyl-1-methylpyrazinium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

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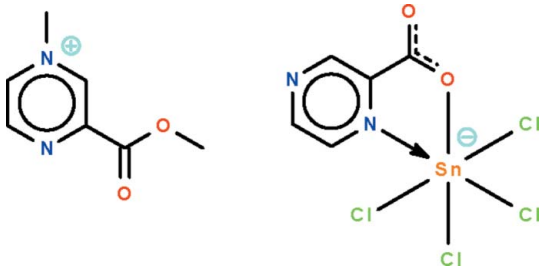
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.025; wR factor = 0.059; data-to-parameter ratio = 17.4.

In the reaction of pyrazine-2-carboxylic acid and stannic chloride in methanol, one equivalent of the carboxylic acid is methylated at the 4-amino site and is also esterified, yielding the title salt, $(C_7H_9N_2O_2)[SnCl_4(C_5H_3N_2O_2)]$. The Sn^{IV} atom in the anion is *N*,*O*-chelated by a pyrazine-2-carboxylate in a *cis*-$SnNOCl_4$ octahedral geometry.

Related literature

For related organotin structures, see: Ma *et al.* (2004).



Experimental

Crystal data

$(C_7H_9N_2O_2)[SnCl_4(C_5H_3N_2O_2)]$
M_r = 536.75
Monoclinic, $P2_1/n$
a = 7.0655 (2) Å
b = 26.7603 (7) Å
c = 9.5220 (2) Å
β = 94.554 (2)°
V = 1794.69 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.05 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent Technologies, 2010)
T_{min} = 0.579, T_{max} = 0.685
8726 measured reflections
3964 independent reflections
3582 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.059
S = 1.02
3964 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent Technologies, 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: SI2325).

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supporting information

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3-Methoxycarbonyl-1-methylpyrazinium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

S1. Comment

The direct synthesis of a potentially chelating amino-carboxylic acid with stannic tetrachloride has not been reported. Pyrazine-2-carboxylic acid yields a number of derivatives with organotin compounds; these are either synthesized by condensing the amino-carboxylic acids with an organotin oxide/hydroxide or by reacting the amino-carboxylic acids with an organotin chloride in the presence of a proton abstractor. With the latter route, the product may be an organostannate in which the pyridine-2-carboxylate chelates to the chlorine-bonded tin atom (Ma *et al.*, 2004). In the reaction of pyrazine-2-carboxylic acid and stannic chloride in methanol, one equivalent of the carboxylic acid is methylated at the 4-amino site and is also esterified to yield the salt, $[C_7H_9N_2O_2]^+$ $[SnCl_4(C_5H_3N_2O_2)]^-$ (Scheme I, Fig. 1). The tin atom in the anion is *N*,*O*-chelated by a pyrazine-2-carboxylate in a *cis*-$SnNOCl_4$ octahedral geometry.

S2. Experimental

Stannic chloride pentahydrate 0.35 g, 1 mmol) and pyrazine-2-carboxylic acid (0.13 g, 1 mmol) were loaded into a convection tube; the tube was filled with dry methanol and kept at 333 K. Colorless crystals were collected from the side arm after several days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

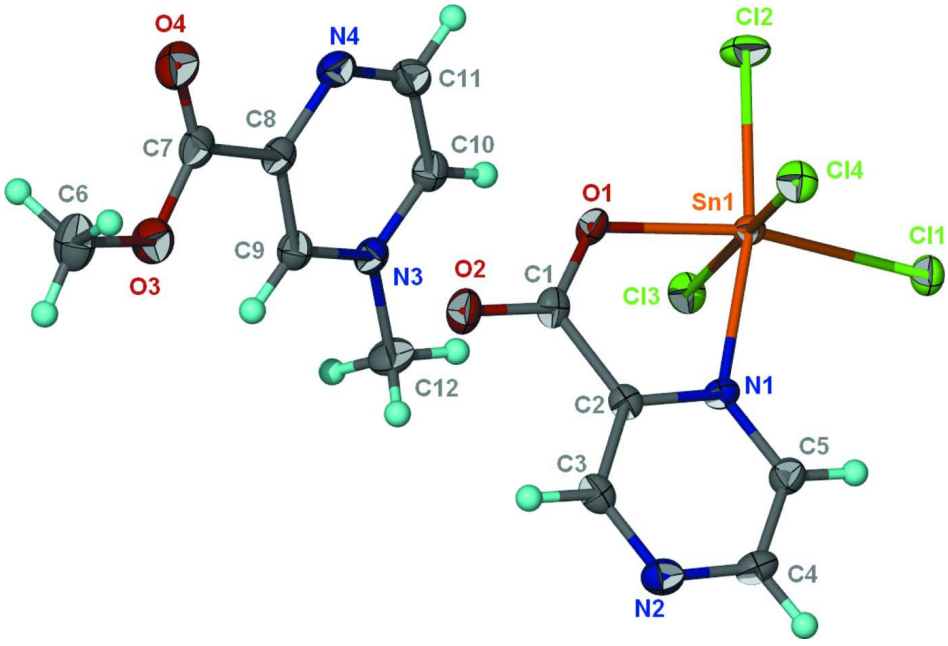

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[C_7H_9N_2O_2]^+$ $[SnCl_4(C_5H_3N_2O_2)]^-$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3-Methoxycarbonyl-1-methylpyrazinium tetrachlorido(pyrazine-2-carboxylato-κ^2N^1,O)stannate(IV)

Crystal data

$(C_7H_9N_2O_2)[SnCl_4(C_5H_3N_2O_2)]$
$M_r = 536.75$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.0655$ (2) Å
$b = 26.7603$ (7) Å
$c = 9.5220$ (2) Å
$\beta = 94.554$ (2)°
$V = 1794.69$ (8) Å^3
$Z = 4$
$F(000) = 1048$
$D_x = 1.987$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5671 reflections
$\theta = 2.3$–$29.2°$
$\mu = 2.05$ mm^{-1}
$T = 100$ K
Prism, colorless
0.30 × 0.25 × 0.20 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 Technologies, 2010)
$T_{min} = 0.579$, $T_{max} = 0.685$
8726 measured reflections
3964 independent reflections
3582 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -7 \rightarrow 9$
$k = -19 \rightarrow 33$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.059$
$S = 1.02$
3964 reflections
228 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 1.1852P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.71$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.60924 (2)	0.655455 (6)	0.329618 (16)	0.01294 (6)
Cl1	0.37306 (8)	0.67760 (2)	0.14686 (6)	0.01817 (13)
Cl2	0.86908 (9)	0.70312 (3)	0.26342 (7)	0.02392 (15)
Cl3	0.50421 (8)	0.72003 (2)	0.48125 (6)	0.01835 (13)
Cl4	0.70745 (9)	0.58382 (2)	0.20077 (6)	0.02003 (14)
O1	0.7650 (2)	0.62593 (7)	0.50542 (17)	0.0159 (4)
O2	0.7745 (3)	0.56931 (7)	0.67716 (18)	0.0218 (4)
O3	1.1503 (3)	0.55738 (7)	1.01670 (19)	0.0237 (4)
O4	1.4080 (3)	0.57009 (8)	0.89617 (19)	0.0265 (4)
N1	0.4045 (3)	0.60323 (8)	0.4241 (2)	0.0137 (4)
N2	0.2115 (3)	0.52822 (8)	0.5541 (2)	0.0189 (5)
N3	0.8601 (3)	0.67062 (8)	0.8236 (2)	0.0157 (4)
N4	1.2150 (3)	0.64576 (8)	0.7473 (2)	0.0194 (5)
C1	0.6920 (3)	0.59043 (9)	0.5772 (2)	0.0151 (5)
C2	0.4893 (3)	0.57708 (9)	0.5319 (2)	0.0135 (5)
C3	0.3911 (4)	0.53973 (9)	0.5957 (2)	0.0167 (5)
H3	0.4537	0.5217	0.6717	0.020*
C4	0.1292 (3)	0.55520 (10)	0.4485 (3)	0.0187 (5)
H4	0.0009	0.5483	0.4172	0.022*
C5	0.2236 (3)	0.59312 (10)	0.3823 (3)	0.0170 (5)
H5	0.1597	0.6117	0.3079	0.020*
C6	1.2439 (4)	0.51458 (11)	1.0871 (3)	0.0283 (6)
H6A	1.1620	0.5007	1.1558	0.042*
H6B	1.3646	0.5253	1.1357	0.042*
H6C	1.2680	0.4890	1.0172	0.042*
C7	1.2504 (4)	0.58058 (10)	0.9240 (3)	0.0190 (5)
C8	1.1371 (3)	0.62241 (10)	0.8531 (2)	0.0161 (5)
C9	0.9594 (3)	0.63438 (10)	0.8935 (3)	0.0171 (5)
H9	0.9082	0.6173	0.9693	0.021*
C10	0.9332 (4)	0.69445 (10)	0.7175 (3)	0.0178 (5)
H10	0.8632	0.7200	0.6675	0.021*
C11	1.1126 (4)	0.68141 (10)	0.6813 (3)	0.0201 (5)
H11	1.1643	0.6987	0.6060	0.024*
C12	0.6682 (3)	0.68313 (11)	0.8634 (3)	0.0225 (6)

H12A	0.6061	0.7056	0.7928	0.034*
H12B	0.6777	0.6996	0.9556	0.034*
H12C	0.5933	0.6525	0.8684	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01198 (9)	0.01295 (10)	0.01393 (9)	−0.00115 (6)	0.00119 (6)	0.00060 (6)
Cl1	0.0190 (3)	0.0184 (3)	0.0166 (3)	0.0002 (2)	−0.0016 (2)	0.0015 (2)
Cl2	0.0176 (3)	0.0224 (4)	0.0324 (3)	−0.0056 (3)	0.0063 (3)	0.0047 (3)
Cl3	0.0208 (3)	0.0170 (3)	0.0172 (3)	0.0022 (2)	0.0015 (2)	−0.0020 (2)
Cl4	0.0217 (3)	0.0188 (3)	0.0199 (3)	0.0023 (2)	0.0029 (2)	−0.0030 (2)
O1	0.0132 (8)	0.0157 (9)	0.0180 (8)	−0.0006 (7)	−0.0036 (7)	0.0014 (7)
O2	0.0225 (9)	0.0220 (11)	0.0200 (9)	−0.0004 (8)	−0.0048 (7)	0.0033 (8)
O3	0.0239 (10)	0.0209 (11)	0.0259 (10)	0.0029 (8)	−0.0005 (8)	0.0048 (8)
O4	0.0220 (10)	0.0319 (12)	0.0253 (10)	0.0089 (8)	0.0009 (8)	−0.0018 (8)
N1	0.0120 (9)	0.0124 (11)	0.0169 (10)	−0.0008 (8)	0.0017 (8)	−0.0009 (8)
N2	0.0182 (10)	0.0157 (12)	0.0230 (11)	−0.0018 (9)	0.0030 (9)	0.0006 (9)
N3	0.0156 (10)	0.0153 (11)	0.0159 (10)	−0.0013 (8)	−0.0009 (8)	−0.0031 (8)
N4	0.0159 (10)	0.0218 (13)	0.0204 (11)	−0.0029 (9)	0.0006 (9)	−0.0014 (9)
C1	0.0188 (12)	0.0119 (13)	0.0143 (11)	0.0015 (10)	−0.0010 (10)	−0.0027 (9)
C2	0.0158 (11)	0.0114 (12)	0.0135 (11)	0.0025 (9)	0.0022 (9)	−0.0007 (9)
C3	0.0223 (12)	0.0133 (13)	0.0147 (11)	0.0003 (10)	0.0025 (10)	−0.0008 (10)
C4	0.0133 (11)	0.0186 (14)	0.0243 (13)	−0.0015 (10)	0.0021 (10)	−0.0018 (11)
C5	0.0154 (11)	0.0171 (14)	0.0183 (12)	0.0007 (10)	0.0002 (10)	0.0006 (10)
C6	0.0344 (16)	0.0207 (16)	0.0282 (14)	0.0040 (12)	−0.0068 (12)	0.0027 (12)
C7	0.0194 (13)	0.0199 (14)	0.0172 (12)	−0.0016 (11)	−0.0025 (10)	−0.0052 (10)
C8	0.0164 (12)	0.0149 (13)	0.0165 (12)	−0.0015 (10)	−0.0012 (10)	−0.0036 (9)
C9	0.0182 (12)	0.0158 (13)	0.0168 (12)	−0.0006 (10)	−0.0010 (10)	−0.0013 (10)
C10	0.0198 (12)	0.0145 (13)	0.0187 (12)	−0.0020 (10)	−0.0019 (10)	−0.0008 (10)
C11	0.0214 (13)	0.0198 (15)	0.0191 (12)	−0.0054 (11)	0.0018 (10)	−0.0004 (11)
C12	0.0147 (12)	0.0246 (16)	0.0284 (14)	0.0036 (11)	0.0037 (11)	−0.0007 (12)

Geometric parameters (Å, º)

Sn1—O1	2.0843 (17)	N4—C8	1.340 (3)
Sn1—N1	2.2499 (19)	C1—C2	1.506 (3)
Sn1—Cl2	2.3619 (6)	C2—C3	1.384 (3)
Sn1—Cl1	2.3881 (6)	C3—H3	0.9500
Sn1—Cl3	2.4065 (6)	C4—C5	1.392 (4)
Sn1—Cl4	2.4076 (6)	C4—H4	0.9500
O1—C1	1.301 (3)	C5—H5	0.9500
O2—C1	1.215 (3)	C6—H6A	0.9800
O3—C7	1.328 (3)	C6—H6B	0.9800
O3—C6	1.459 (3)	C6—H6C	0.9800
O4—C7	1.199 (3)	C7—C8	1.504 (4)
N1—C5	1.336 (3)	C8—C9	1.380 (3)
N1—C2	1.343 (3)	C9—H9	0.9500

N2—C4	1.333 (3)	C10—C11	1.384 (4)
N2—C3	1.335 (3)	C10—H10	0.9500
N3—C10	1.333 (3)	C11—H11	0.9500
N3—C9	1.342 (3)	C12—H12A	0.9800
N3—C12	1.475 (3)	C12—H12B	0.9800
N4—C11	1.326 (3)	C12—H12C	0.9800

O1—Sn1—N1	76.03 (7)	N2—C4—C5	122.7 (2)
O1—Sn1—Cl2	92.70 (5)	N2—C4—H4	118.6
N1—Sn1—Cl2	168.60 (5)	C5—C4—H4	118.6
O1—Sn1—Cl1	166.67 (5)	N1—C5—C4	119.6 (2)
N1—Sn1—Cl1	90.65 (5)	N1—C5—H5	120.2
Cl2—Sn1—Cl1	100.63 (2)	C4—C5—H5	120.2
O1—Sn1—Cl3	87.62 (5)	O3—C6—H6A	109.5
N1—Sn1—Cl3	88.13 (5)	O3—C6—H6B	109.5
Cl2—Sn1—Cl3	93.19 (2)	H6A—C6—H6B	109.5
Cl1—Sn1—Cl3	91.61 (2)	O3—C6—H6C	109.5
O1—Sn1—Cl4	87.27 (5)	H6A—C6—H6C	109.5
N1—Sn1—Cl4	85.97 (5)	H6B—C6—H6C	109.5
Cl2—Sn1—Cl4	91.86 (2)	O4—C7—O3	126.2 (3)
Cl1—Sn1—Cl4	92.25 (2)	O4—C7—C8	123.1 (2)
Cl3—Sn1—Cl4	172.98 (2)	O3—C7—C8	110.7 (2)
C1—O1—Sn1	119.54 (15)	N4—C8—C9	122.6 (2)
C7—O3—C6	115.3 (2)	N4—C8—C7	116.6 (2)
C5—N1—C2	118.7 (2)	C9—C8—C7	120.8 (2)
C5—N1—Sn1	129.58 (17)	N3—C9—C8	118.7 (2)
C2—N1—Sn1	111.54 (15)	N3—C9—H9	120.6
C4—N2—C3	116.5 (2)	C8—C9—H9	120.6
C10—N3—C9	120.2 (2)	N3—C10—C11	119.1 (2)
C10—N3—C12	120.4 (2)	N3—C10—H10	120.5
C9—N3—C12	119.4 (2)	C11—C10—H10	120.5
C11—N4—C8	116.8 (2)	N4—C11—C10	122.6 (2)
O2—C1—O1	124.5 (2)	N4—C11—H11	118.7
O2—C1—C2	120.0 (2)	C10—C11—H11	118.7
O1—C1—C2	115.5 (2)	N3—C12—H12A	109.5
N1—C2—C3	120.2 (2)	N3—C12—H12B	109.5
N1—C2—C1	116.9 (2)	H12A—C12—H12B	109.5
C3—C2—C1	122.9 (2)	N3—C12—H12C	109.5
N2—C3—C2	122.3 (2)	H12A—C12—H12C	109.5
N2—C3—H3	118.9	H12B—C12—H12C	109.5
C2—C3—H3	118.9		

N1—Sn1—O1—C1	6.32 (17)	O1—C1—C2—C3	179.1 (2)
Cl2—Sn1—O1—C1	−171.93 (17)	C4—N2—C3—C2	−1.1 (4)
Cl1—Sn1—O1—C1	8.0 (3)	N1—C2—C3—N2	0.0 (4)
Cl3—Sn1—O1—C1	94.98 (17)	C1—C2—C3—N2	−179.8 (2)
Cl4—Sn1—O1—C1	−80.20 (17)	C3—N2—C4—C5	0.8 (4)
O1—Sn1—N1—C5	178.9 (2)	C2—N1—C5—C4	−1.6 (4)

Cl2—Sn1—N1—C5	−172.28 (19)	Sn1—N1—C5—C4	173.07 (17)
Cl1—Sn1—N1—C5	−0.7 (2)	N2—C4—C5—N1	0.5 (4)
Cl3—Sn1—N1—C5	90.9 (2)	C6—O3—C7—O4	−1.4 (4)
Cl4—Sn1—N1—C5	−92.9 (2)	C6—O3—C7—C8	177.4 (2)
O1—Sn1—N1—C2	−6.18 (15)	C11—N4—C8—C9	−0.2 (4)
Cl2—Sn1—N1—C2	2.7 (4)	C11—N4—C8—C7	177.6 (2)
Cl1—Sn1—N1—C2	174.22 (15)	O4—C7—C8—N4	5.1 (4)
Cl3—Sn1—N1—C2	−94.20 (15)	O3—C7—C8—N4	−173.7 (2)
Cl4—Sn1—N1—C2	82.01 (15)	O4—C7—C8—C9	−177.1 (2)
Sn1—O1—C1—O2	176.53 (19)	O3—C7—C8—C9	4.1 (3)
Sn1—O1—C1—C2	−5.3 (3)	C10—N3—C9—C8	−0.1 (4)
C5—N1—C2—C3	1.4 (3)	C12—N3—C9—C8	178.7 (2)
Sn1—N1—C2—C3	−174.21 (18)	N4—C8—C9—N3	0.4 (4)
C5—N1—C2—C1	−178.8 (2)	C7—C8—C9—N3	−177.3 (2)
Sn1—N1—C2—C1	5.6 (3)	C9—N3—C10—C11	−0.3 (4)
O2—C1—C2—N1	177.5 (2)	C12—N3—C10—C11	−179.1 (2)
O1—C1—C2—N1	−0.7 (3)	C8—N4—C11—C10	−0.2 (4)
O2—C1—C2—C3	−2.7 (4)	N3—C10—C11—N4	0.5 (4)