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4-(Dimethylamino)pyridinium tetrachlorido(pyridine-2-carboxylato-κ^2N,O)-stannate(IV)

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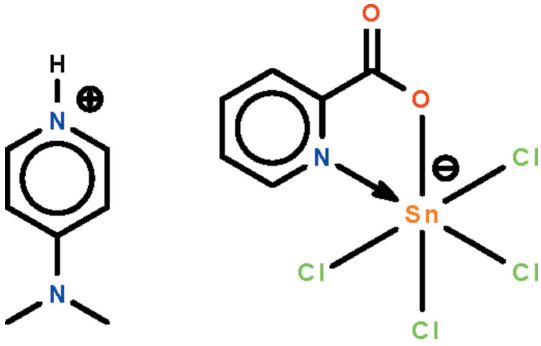
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.020; wR factor = 0.049; data-to-parameter ratio = 19.2.

The reaction of 4-(dimethylamino)pyridine, picolinic acid and stannic chloride yields the title salt, $(C_7H_{11}N_2)[SnCl_4(C_6H_4NO_2)]$, in which the Sn^{IV} atom is N,O-chelated by the picolinate ion in a *cis*-$SnNOCl_4$ octahedral geometry. The cation is linked to the anion by an N—H···O hydrogen bond.

Related literature

For 4-(dimethylamino)pyridinium tetrachlorido(quinoline-2-carboxylato)stannate, see: Najafi *et al.* (2011).



Experimental

Crystal data

$(C_7H_{11}N_2)[SnCl_4(C_6H_4NO_2)]$
M_r = 505.77
Triclinic, $P\overline{1}$
a = 7.6658 (2) Å
b = 9.8948 (4) Å
c = 13.5722 (6) Å
α = 69.485 (4)°
β = 83.159 (3)°
γ = 67.900 (3)°
V = 893.25 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.04 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, 2012)
T_{min} = 0.580, T_{max} = 0.686
12966 measured reflections
4109 independent reflections
3744 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.049
S = 1.02
4109 reflections
214 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.87 (1)	1.93 (1)	2.802 (2)	176 (2)

Data collection: *CrysAlis PRO* (Agilent, 2012); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5909).

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supplementary materials

Acta Cryst. (2012). E**68**, m744 [doi:10.1107/S160053681201968X]

4-(Dimethylamino)pyridinium tetrachlorido(pyridine-2-carboxylato-κ^2N,O)stannate(IV)

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

Comment

A previous study reported 4-(dimethylamino)pyridinium tetrachlorido(quinoline-2-carboxylato)stannate, which was synthesized by the reaction of 4-(dimethylamino)pyridine, quinoline-2-carboxylic acid and stannic chloride in methanol (Najafi *et al.*, 2011). The reaction with picolinic acid in place of quinoline-2-carboxylic acid yielded the analogous salt, $(C_7H_{11}N_2)[SnCl_4(C_6H_4NO_2)]$ (Scheme I). The Sn^{IV} atom is *N*,*O*-chelated by the picolinate ion in a *cis*-$SnNOCl_4$ octahedral geometry (Fig. 1). The cation is linked to the anion by an N–H···O hydrogen bond (Table 1).

Experimental

Stannic chloride pentahydrate (0.35 g, 1 mmol), picolinic acid (0.12 g, 1 mmol) and 4-(dimethylamino)pyridine (0.12 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.

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.

The pyridinium H-atom was located in a difference Fourier map, and was refined isotropically with a distance restraint of N–H 0.88±0.01 Å.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); 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).

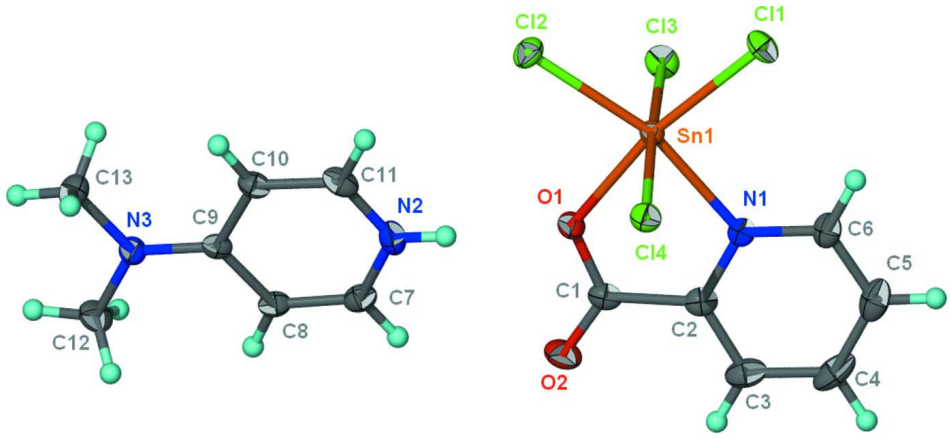

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $(C_7H_{11}N_2)[SnCl_4(C_6H_4NO_2)]$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-(Dimethylamino)pyridinium tetrachlorido(pyridine-2-carboxylato-κ^2N,O)stannate(IV)

Crystal data

$(C_7H_{11}N_2)[SnCl_4(C_6H_4NO_2)]$
$M_r = 505.77$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.6658$ (2) Å
$b = 9.8948$ (4) Å
$c = 13.5722$ (6) Å
$\alpha = 69.485$ (4)°
$\beta = 83.159$ (3)°
$\gamma = 67.900$ (3)°
$V = 893.25$ (6) Å^3
$Z = 2$
$F(000) = 496$
$D_x = 1.880$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8614 reflections
$\theta = 2.4$–$27.5°$
$\mu = 2.04$ 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 scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.580$, $T_{max} = 0.686$
12966 measured reflections
4109 independent reflections
3744 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.4°$
$h = -9\rightarrow9$
$k = -12\rightarrow12$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.049$
$S = 1.02$
4109 reflections
214 parameters
1 restraint
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

$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 0.1127P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.471694 (16)	0.931155 (14)	0.787934 (10)	0.01225 (5)
Cl1	0.41910 (7)	1.16043 (5)	0.82535 (4)	0.01887 (11)
Cl2	0.63070 (7)	0.97801 (6)	0.62519 (4)	0.01884 (11)
Cl3	0.76267 (6)	0.79583 (6)	0.88640 (4)	0.01957 (11)
Cl4	0.16639 (6)	1.04910 (5)	0.70014 (4)	0.01687 (10)
O1	0.48413 (18)	0.72139 (15)	0.77597 (11)	0.0164 (3)
O2	0.3917 (2)	0.51987 (16)	0.84279 (12)	0.0231 (3)
N1	0.3269 (2)	0.83176 (18)	0.93069 (12)	0.0137 (3)
N2	0.6230 (2)	0.5870 (2)	0.61853 (14)	0.0184 (4)
H2	0.574 (3)	0.630 (3)	0.6665 (15)	0.038 (7)*
N3	0.8688 (2)	0.35910 (18)	0.40725 (13)	0.0153 (3)
C1	0.3977 (3)	0.6389 (2)	0.84580 (16)	0.0159 (4)
C2	0.3057 (3)	0.7014 (2)	0.93305 (15)	0.0155 (4)
C3	0.2055 (3)	0.6298 (2)	1.01141 (17)	0.0217 (5)
H3	0.1910	0.5382	1.0122	0.026*
C4	0.1268 (3)	0.6952 (3)	1.08887 (17)	0.0270 (5)
H4	0.0545	0.6500	1.1428	0.032*
C5	0.1535 (3)	0.8254 (3)	1.08742 (16)	0.0235 (5)
H5	0.1024	0.8694	1.1412	0.028*
C6	0.2552 (3)	0.8922 (2)	1.00731 (15)	0.0178 (4)
H6	0.2744	0.9819	1.0066	0.021*
C7	0.6389 (3)	0.4385 (2)	0.64189 (16)	0.0176 (4)
H7	0.5927	0.3883	0.7070	0.021*
C8	0.7198 (2)	0.3592 (2)	0.57414 (15)	0.0153 (4)
H8	0.7304	0.2546	0.5922	0.018*
C9	0.7888 (2)	0.4335 (2)	0.47584 (15)	0.0137 (4)
C10	0.7676 (3)	0.5896 (2)	0.45468 (16)	0.0159 (4)
H10	0.8102	0.6444	0.3900	0.019*
C11	0.6865 (3)	0.6614 (2)	0.52659 (17)	0.0183 (4)
H11	0.6745	0.7657	0.5117	0.022*
C12	0.8845 (3)	0.1993 (2)	0.42939 (17)	0.0213 (5)
H12A	0.9601	0.1329	0.4937	0.032*
H12B	0.9454	0.1647	0.3704	0.032*
H12C	0.7585	0.1933	0.4390	0.032*
C13	0.9392 (3)	0.4362 (2)	0.30663 (16)	0.0201 (4)
H13A	1.0157	0.4893	0.3186	0.030*
H13B	0.8327	0.5118	0.2599	0.030*
H13C	1.0164	0.3592	0.2741	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01423 (8)	0.01017 (8)	0.01266 (8)	−0.00471 (5)	0.00094 (5)	−0.00403 (5)

Cl1	0.0237 (2)	0.0125 (2)	0.0218 (3)	−0.00547 (19)	−0.00284 (19)	−0.0074 (2)
Cl2	0.0212 (2)	0.0168 (2)	0.0171 (3)	−0.00816 (19)	0.00602 (18)	−0.00436 (19)
Cl3	0.0165 (2)	0.0175 (2)	0.0221 (3)	−0.00282 (19)	−0.00390 (18)	−0.0058 (2)
Cl4	0.0166 (2)	0.0169 (2)	0.0167 (2)	−0.00586 (19)	−0.00196 (17)	−0.00459 (19)
O1	0.0222 (7)	0.0122 (7)	0.0173 (7)	−0.0088 (6)	0.0041 (5)	−0.0062 (6)
O2	0.0294 (8)	0.0166 (8)	0.0270 (9)	−0.0130 (6)	0.0004 (6)	−0.0063 (6)
N1	0.0137 (8)	0.0129 (8)	0.0121 (8)	−0.0034 (6)	−0.0013 (6)	−0.0025 (7)
N2	0.0170 (8)	0.0200 (9)	0.0193 (9)	−0.0035 (7)	−0.0013 (7)	−0.0105 (8)
N3	0.0178 (8)	0.0118 (8)	0.0167 (9)	−0.0054 (7)	0.0020 (6)	−0.0054 (7)
C1	0.0149 (9)	0.0134 (10)	0.0178 (10)	−0.0052 (8)	−0.0041 (7)	−0.0019 (8)
C2	0.0136 (9)	0.0131 (10)	0.0156 (10)	−0.0032 (8)	−0.0040 (7)	−0.0001 (8)
C3	0.0189 (10)	0.0203 (11)	0.0214 (11)	−0.0093 (8)	−0.0026 (8)	0.0020 (9)
C4	0.0186 (10)	0.0331 (13)	0.0181 (12)	−0.0088 (9)	0.0027 (8)	0.0032 (10)
C5	0.0190 (10)	0.0286 (12)	0.0143 (11)	−0.0009 (9)	−0.0011 (8)	−0.0050 (9)
C6	0.0168 (10)	0.0197 (11)	0.0133 (10)	−0.0028 (8)	−0.0017 (7)	−0.0047 (8)
C7	0.0144 (9)	0.0201 (11)	0.0168 (10)	−0.0071 (8)	−0.0009 (7)	−0.0031 (8)
C8	0.0138 (9)	0.0125 (10)	0.0177 (10)	−0.0052 (8)	−0.0010 (7)	−0.0018 (8)
C9	0.0105 (9)	0.0130 (10)	0.0165 (10)	−0.0035 (7)	−0.0027 (7)	−0.0036 (8)
C10	0.0174 (9)	0.0120 (10)	0.0179 (10)	−0.0060 (8)	−0.0029 (7)	−0.0027 (8)
C11	0.0177 (10)	0.0120 (10)	0.0244 (11)	−0.0036 (8)	−0.0062 (8)	−0.0048 (8)
C12	0.0227 (10)	0.0138 (10)	0.0289 (12)	−0.0058 (8)	0.0011 (9)	−0.0099 (9)
C13	0.0208 (10)	0.0208 (11)	0.0170 (11)	−0.0067 (9)	0.0030 (8)	−0.0059 (9)

Geometric parameters (Å, º)

Sn1—O1	2.1041 (13)	C4—C5	1.372 (3)
Sn1—N1	2.2194 (16)	C4—H4	0.9500
Sn1—Cl1	2.3743 (5)	C5—C6	1.381 (3)
Sn1—Cl2	2.3761 (5)	C5—H5	0.9500
Sn1—Cl3	2.4006 (5)	C6—H6	0.9500
Sn1—Cl4	2.4277 (5)	C7—C8	1.359 (3)
O1—C1	1.311 (2)	C7—H7	0.9500
O2—C1	1.210 (2)	C8—C9	1.427 (3)
N1—C6	1.339 (2)	C8—H8	0.9500
N1—C2	1.348 (3)	C9—C10	1.416 (3)
N2—C11	1.348 (3)	C10—C11	1.361 (3)
N2—C7	1.350 (3)	C10—H10	0.9500
N2—H2	0.874 (10)	C11—H11	0.9500
N3—C9	1.338 (2)	C12—H12A	0.9800
N3—C12	1.462 (3)	C12—H12B	0.9800
N3—C13	1.464 (2)	C12—H12C	0.9800
C1—C2	1.506 (3)	C13—H13A	0.9800
C2—C3	1.382 (3)	C13—H13B	0.9800
C3—C4	1.386 (3)	C13—H13C	0.9800
C3—H3	0.9500		
O1—Sn1—N1	75.84 (6)	C5—C4—H4	120.1
O1—Sn1—Cl1	169.89 (4)	C3—C4—H4	120.1
N1—Sn1—Cl1	94.27 (4)	C4—C5—C6	119.6 (2)
O1—Sn1—Cl2	88.75 (4)	C4—C5—H5	120.2

N1—Sn1—Cl2	164.57 (4)	C6—C5—H5	120.2
Cl1—Sn1—Cl2	101.090 (18)	N1—C6—C5	120.7 (2)
O1—Sn1—Cl3	89.31 (4)	N1—C6—H6	119.6
N1—Sn1—Cl3	88.57 (4)	C5—C6—H6	119.6
Cl1—Sn1—Cl3	92.581 (18)	N2—C7—C8	121.32 (19)
Cl2—Sn1—Cl3	92.190 (18)	N2—C7—H7	119.3
O1—Sn1—Cl4	87.20 (4)	C8—C7—H7	119.3
N1—Sn1—Cl4	86.38 (4)	C7—C8—C9	119.91 (19)
Cl1—Sn1—Cl4	90.124 (17)	C7—C8—H8	120.0
Cl2—Sn1—Cl4	92.055 (17)	C9—C8—H8	120.0
Cl3—Sn1—Cl4	174.439 (17)	N3—C9—C10	121.88 (18)
C1—O1—Sn1	119.22 (12)	N3—C9—C8	121.49 (18)
C6—N1—C2	120.02 (17)	C10—C9—C8	116.63 (18)
C6—N1—Sn1	126.65 (14)	C11—C10—C9	120.29 (19)
C2—N1—Sn1	113.24 (12)	C11—C10—H10	119.9
C11—N2—C7	120.66 (18)	C9—C10—H10	119.9
C11—N2—H2	122.3 (18)	N2—C11—C10	121.18 (19)
C7—N2—H2	117.0 (18)	N2—C11—H11	119.4
C9—N3—C12	120.98 (17)	C10—C11—H11	119.4
C9—N3—C13	121.09 (17)	N3—C12—H12A	109.5
C12—N3—C13	117.91 (16)	N3—C12—H12B	109.5
O2—C1—O1	123.64 (18)	H12A—C12—H12B	109.5
O2—C1—C2	121.19 (18)	N3—C12—H12C	109.5
O1—C1—C2	115.16 (17)	H12A—C12—H12C	109.5
N1—C2—C3	121.61 (19)	H12B—C12—H12C	109.5
N1—C2—C1	116.16 (17)	N3—C13—H13A	109.5
C3—C2—C1	122.22 (19)	N3—C13—H13B	109.5
C2—C3—C4	118.2 (2)	H13A—C13—H13B	109.5
C2—C3—H3	120.9	N3—C13—H13C	109.5
C4—C3—H3	120.9	H13A—C13—H13C	109.5
C5—C4—C3	119.8 (2)	H13B—C13—H13C	109.5

N1—Sn1—O1—C1	4.83 (13)	O1—C1—C2—N1	−1.6 (2)
Cl1—Sn1—O1—C1	−7.3 (3)	O2—C1—C2—C3	−2.2 (3)
Cl2—Sn1—O1—C1	−174.26 (13)	O1—C1—C2—C3	178.52 (17)
Cl3—Sn1—O1—C1	93.53 (13)	N1—C2—C3—C4	−0.3 (3)
Cl4—Sn1—O1—C1	−82.14 (13)	C1—C2—C3—C4	179.61 (17)
O1—Sn1—N1—C6	178.01 (16)	C2—C3—C4—C5	−1.5 (3)
Cl1—Sn1—N1—C6	−4.12 (15)	C3—C4—C5—C6	1.5 (3)
Cl2—Sn1—N1—C6	−178.59 (11)	C2—N1—C6—C5	−2.2 (3)
Cl3—Sn1—N1—C6	88.36 (15)	Sn1—N1—C6—C5	174.16 (13)
Cl4—Sn1—N1—C6	−93.97 (15)	C4—C5—C6—N1	0.4 (3)
O1—Sn1—N1—C2	−5.40 (12)	C11—N2—C7—C8	0.3 (3)
Cl1—Sn1—N1—C2	172.47 (12)	N2—C7—C8—C9	−0.4 (3)
Cl2—Sn1—N1—C2	−2.0 (2)	C12—N3—C9—C10	−178.27 (17)
Cl3—Sn1—N1—C2	−95.05 (12)	C13—N3—C9—C10	0.2 (3)
Cl4—Sn1—N1—C2	82.62 (12)	C12—N3—C9—C8	1.5 (3)
Sn1—O1—C1—O2	177.24 (15)	C13—N3—C9—C8	−179.96 (16)
Sn1—O1—C1—C2	−3.5 (2)	C7—C8—C9—N3	−179.87 (17)

C6—N1—C2—C3	2.2 (3)	C7—C8—C9—C10	−0.1 (3)
Sn1—N1—C2—C3	−174.69 (14)	N3—C9—C10—C11	−179.60 (17)
C6—N1—C2—C1	−177.72 (16)	C8—C9—C10—C11	0.6 (3)
Sn1—N1—C2—C1	5.44 (19)	C7—N2—C11—C10	0.3 (3)
O2—C1—C2—N1	177.67 (17)	C9—C10—C11—N2	−0.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.87 (1)	1.93 (1)	2.802 (2)	176 (2)