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catena-Poly[[aquadiphenyltin(IV)]-μ-2,6-pyridinedicarboxylato]

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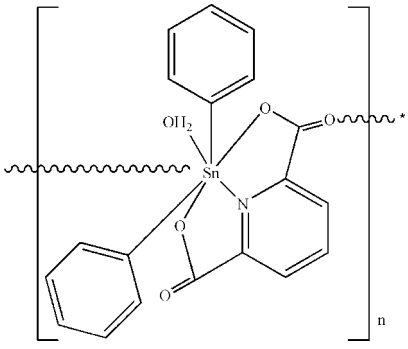
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 13.6.

In the title compound, $[Sn(C_6H_5)_2(C_7H_3NO_4)(H_2O)]_n$, two phenyl and one 2,6-pyridinedicarboxylate group and one water molecule are bonded to the Sn atom in a distorted pentagonal–bipyramidal geometry; ; the phenyl groups are in axial positions, and one N and three O atoms of 2,6-pyridinedicarboxylate and one water O atom are in equatorial positions. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules.

Related literature

For general background, see: Gielen *et al.* (1988). For related literature, see: Ng (1999). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$[Sn(C_6H_5)_2(C_7H_3NO_4)(H_2O)]$
M_r = 456.03
Monoclinic, $P2_1/c$
a = 9.779 (2) Å
b = 9.807 (2) Å
c = 19.334 (4) Å
β = 100.915 (3)°
V = 1820.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.43 mm^{-1}
T = 298 (2) K
0.48 × 0.45 × 0.44 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.509, T_{max} = 0.533
9237 measured reflections
3206 independent reflections
2651 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.03
3206 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −1.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.222 (3)	Sn1—N1	2.342 (4)
Sn1—O3	2.449 (3)	Sn1—C8	2.108 (6)
Sn1—O4^{i}	2.384 (3)	Sn1—C14	2.123 (6)
Sn1—O5	2.268 (3)		
O1—Sn1—O3	135.61 (12)	C8—Sn1—O3	87.9 (2)
O1—Sn1—O4^{i}	151.67 (13)	C8—Sn1—O4^{i}	86.6 (2)
O1—Sn1—O5	73.54 (12)	C8—Sn1—O5	87.1 (2)
O4^{i}—Sn1—O3	72.62 (12)	C8—Sn1—N1	90.43 (19)
O5—Sn1—O3	150.79 (12)	C8—Sn1—C14	172.6 (2)
O5—Sn1—O4^{i}	78.37 (12)	C14—Sn1—O1	91.42 (19)
O1—Sn1—N1	69.74 (13)	C14—Sn1—O3	88.95 (19)
O5—Sn1—N1	142.76 (13)	C14—Sn1—O4^{i}	86.09 (18)
N1—Sn1—O3	65.98 (12)	C14—Sn1—O5	92.49 (18)
N1—Sn1—O4^{i}	138.57 (12)	C14—Sn1—N1	94.38 (18)
C8—Sn1—O1	95.5 (2)		

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1···O2^{ii}	0.83	1.85	2.671 (3)	167
O5—H2···O3^{i}	0.95	1.74	2.673 (3)	166

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

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2372).

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supplementary materials

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catena-Poly[[aquadiphenyltin(IV)]-μ-2,6-pyridinedicarboxylato]

F. Li, Y. Li, H. Zhao and X. Lang

Comment

Self-assembled organotin derivatives of carboxylic acid ligands have been extensively studied due to their biological activities (Gielen *et al.*, 1988). Bi- or multidentate ligands containing O– or N-donors are often used to coordinate to tin centers. 2,6-pyridinedicarboxylic acid is a good bridging ligand that can sometimes be used to generate unexpected and interesting coordination polymers. We report herein the crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1), contains two phenyl and one (2,6-pyridinedicarboxylate) groups bonded to the tin atom, where the bond lengths and angles (Table 1) are generally within normal ranges (Allen *et al.*, 1987). The tin atom has a distorted pentagonal bipyramidal geometry with atoms C8 and C14 of the phenyl groups in axial positions and N1, O1, O3 and O4^{i} atoms of 2,6-pyridinedicarboxylic acid [symmetry code: (i) $-x + 1$, $y - 1/2$, $-z + 1/2$] and O5 atom of water molecule in equatorial position, as in the similar compound, bis(dicyclohexylammonium)(oxalato)(pyridine-2,6-dicarboxyl- ato)dibutylstannate 3.5 hydrate (Ng, 1999).

The phenyl rings A (C8—C13) and B (C14—C19) are, of course, planar and the dihedral angle between them is A/B = 16.67 (3)°. The ring system containing the 2,6-pyridinedicarboxylic acid group C (Sn1/O1/O3/N1/C1—C7) is nearly planar and it is oriented with respect to the phenyl rings at dihedral angles of A/C = 87.11 (3)° and B/C = 84.38 (2)°.

In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 2, Fig. 2) link the molecules, in which they seem to be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, a mixture of diphenyltin dichloride (688 mg, 2.0 mmol), 2,6-pyridinedicarboxylic acid (334 mg, 2.0 mmol) and sodium ethoxide (272 mg, 4.0 mmol) in methanol with water (80 ml) was heated under reflux for 14 h at 303 K. The resulting clear solution was evaporated under vacuum and the product recrystallized from a mixture of dichloromethane-hexane (1:1) to yield colorless, block-like crystals of (I) (yield; 777 mg, 76%, m.p. 445 K). Analysis; calculated for $C_{38}H_{30}N_2O_{10}Sn2$: C 50.04, H, 3.32; N 3.07%; found: C 50.12, H 3.46, N, 3.01%.

Refinement

H atoms (for H_2O) wer located in difference syntheses and constrained to ride on their parent atom [O—H = 0.8347 and 0.9480 Å and $U_{iso}(H) = 1.2U_{eq}(O)$]. The remaining H atoms were positioned geometrically, with C—H = 0.93 Å, for aromatic H atoms and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

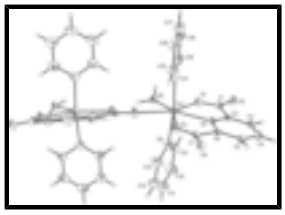

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

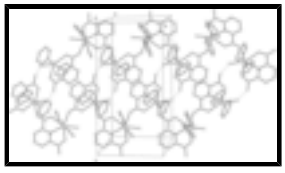

Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines. The non-bonding H atoms have been omitted for clarity.

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Crystal data

$[Sn(C_6H_5)_2(C_7H_3NO_4)(H_2O)]$	F_{000} = 904
M_r = 456.03	D_x = 1.664 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4957 reflections
a = 9.779 (2) Å	θ = 2.3–28.2º
b = 9.807 (2) Å	μ = 1.43 mm^{-1}
c = 19.334 (4) Å	T = 298 (2) K
β = 100.915 (3)º	Block, colorless
V = 1820.6 (6) Å^3	0.48 × 0.45 × 0.44 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3206 independent reflections
Radiation source: fine-focus sealed tube	2651 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −11→11
T_{min} = 0.509, T_{max} = 0.533	k = −11→11
9237 measured reflections	l = −12→22

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.094$	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 4.0544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
3206 reflections	$\Delta\rho_{max} = 0.72$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -1.33$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.52733 (3)	0.67363 (3)	0.156159 (16)	0.02997 (14)
O1	0.5801 (4)	0.6306 (3)	0.05146 (18)	0.0376 (8)
O2	0.6472 (4)	0.7069 (4)	−0.04578 (19)	0.0458 (9)
O3	0.5119 (4)	0.8781 (3)	0.22701 (18)	0.0392 (8)
O4	0.5509 (4)	1.1034 (3)	0.23984 (18)	0.0399 (9)
O5	0.5175 (4)	0.4439 (3)	0.14243 (18)	0.0398 (9)
H1	0.4618	0.4086	0.1092	0.048*
H2	0.5031	0.4064	0.1857	0.048*
N1	0.5961 (4)	0.8744 (4)	0.10674 (19)	0.0275 (8)
C1	0.6218 (5)	0.7222 (5)	0.0138 (3)	0.0336 (11)
C2	0.6398 (5)	0.8629 (5)	0.0454 (2)	0.0290 (10)
C3	0.6923 (5)	0.9729 (5)	0.0147 (3)	0.0383 (12)
H3	0.7229	0.9635	−0.0277	0.046*
C4	0.6983 (6)	1.0963 (6)	0.0480 (3)	0.0427 (13)
H4	0.7351	1.1715	0.0286	0.051*
C5	0.6502 (5)	1.1100 (5)	0.1103 (3)	0.0375 (12)
H5	0.6512	1.1943	0.1324	0.045*
C6	0.6004 (5)	0.9949 (4)	0.1391 (2)	0.0288 (10)
C7	0.5501 (5)	0.9926 (5)	0.2079 (2)	0.0317 (11)
C8	0.7311 (6)	0.6460 (6)	0.2137 (4)	0.0541 (13)
C9	0.8249 (7)	0.5751 (8)	0.1817 (4)	0.0738 (15)
H9	0.7960	0.5409	0.1364	0.089*
C10	0.9632 (8)	0.5539 (9)	0.2166 (5)	0.0842 (16)
H10	1.0270	0.5071	0.1954	0.101*

C11	0.9987 (9)	0.6058 (10)	0.2832 (5)	0.0907 (18)
H11	1.0907	0.5963	0.3062	0.109*
C12	0.9095 (9)	0.6702 (8)	0.3182 (5)	0.0861 (17)
H12	0.9382	0.6996	0.3644	0.103*
C13	0.7720 (8)	0.6909 (7)	0.2821 (4)	0.0730 (15)
H13	0.7084	0.7352	0.3045	0.088*
C14	0.3134 (6)	0.6972 (6)	0.1108 (3)	0.0444 (11)
C15	0.2267 (7)	0.7880 (7)	0.1361 (4)	0.0599 (13)
H15	0.2600	0.8396	0.1761	0.072*
C16	0.0885 (7)	0.8016 (8)	0.1012 (4)	0.0685 (14)
H16	0.0311	0.8647	0.1175	0.082*
C17	0.0364 (7)	0.7242 (8)	0.0437 (4)	0.0668 (15)
H17	−0.0560	0.7338	0.0213	0.080*
C18	0.1205 (7)	0.6325 (7)	0.0192 (4)	0.0591 (13)
H18	0.0851	0.5787	−0.0197	0.071*
C19	0.2583 (6)	0.6191 (6)	0.0519 (3)	0.0517 (12)
H19	0.3150	0.5570	0.0344	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0422 (2)	0.0238 (2)	0.0241 (2)	−0.00080 (14)	0.00672 (14)	−0.00005 (13)
O1	0.059 (2)	0.0259 (17)	0.0308 (19)	−0.0052 (16)	0.0157 (17)	−0.0059 (15)
O2	0.066 (3)	0.044 (2)	0.031 (2)	−0.0080 (18)	0.0198 (18)	−0.0076 (17)
O3	0.068 (2)	0.0231 (17)	0.0297 (18)	−0.0052 (16)	0.0177 (17)	−0.0014 (15)
O4	0.066 (2)	0.0250 (18)	0.0322 (19)	−0.0047 (16)	0.0177 (17)	−0.0055 (15)
O5	0.065 (2)	0.0246 (18)	0.0302 (19)	−0.0053 (16)	0.0104 (17)	−0.0008 (14)
N1	0.036 (2)	0.025 (2)	0.021 (2)	−0.0011 (16)	0.0051 (16)	−0.0031 (16)
C1	0.040 (3)	0.033 (3)	0.028 (3)	−0.001 (2)	0.007 (2)	−0.005 (2)
C2	0.032 (2)	0.031 (2)	0.023 (2)	0.0004 (19)	0.0036 (19)	0.0002 (19)
C3	0.048 (3)	0.038 (3)	0.031 (3)	−0.001 (2)	0.014 (2)	0.004 (2)
C4	0.053 (3)	0.035 (3)	0.042 (3)	−0.007 (2)	0.013 (3)	0.009 (2)
C5	0.049 (3)	0.024 (3)	0.037 (3)	−0.001 (2)	0.004 (2)	−0.002 (2)
C6	0.037 (3)	0.021 (2)	0.026 (2)	0.0008 (19)	−0.0003 (19)	−0.0002 (19)
C7	0.042 (3)	0.024 (3)	0.027 (2)	0.001 (2)	0.004 (2)	−0.001 (2)
C8	0.049 (2)	0.054 (3)	0.055 (3)	−0.003 (2)	0.000 (2)	0.008 (2)
C9	0.062 (3)	0.083 (3)	0.072 (3)	0.005 (3)	0.002 (3)	0.009 (3)
C10	0.069 (3)	0.091 (3)	0.088 (3)	0.009 (3)	0.003 (3)	0.007 (3)
C11	0.072 (3)	0.094 (3)	0.095 (3)	−0.004 (3)	−0.014 (3)	0.005 (3)
C12	0.083 (3)	0.084 (3)	0.079 (3)	−0.004 (3)	−0.018 (3)	−0.002 (3)
C13	0.068 (3)	0.070 (3)	0.069 (3)	0.000 (3)	−0.015 (3)	0.000 (3)
C14	0.043 (2)	0.047 (2)	0.044 (2)	−0.007 (2)	0.010 (2)	0.007 (2)
C15	0.053 (3)	0.064 (3)	0.063 (3)	0.000 (2)	0.010 (2)	−0.004 (2)
C16	0.057 (3)	0.074 (3)	0.073 (3)	0.007 (3)	0.012 (3)	−0.002 (3)
C17	0.051 (3)	0.073 (3)	0.072 (3)	−0.007 (3)	0.003 (3)	0.007 (3)
C18	0.056 (3)	0.057 (3)	0.059 (3)	−0.012 (2)	−0.002 (2)	0.007 (2)
C19	0.051 (2)	0.047 (2)	0.054 (3)	−0.008 (2)	0.000 (2)	0.006 (2)

Geometric parameters (Å, °)

Sn1—O1	2.222 (3)	C6—C7	1.503 (7)
Sn1—O3	2.449 (3)	C8—C13	1.380 (10)
Sn1—O4^{i}	2.384 (3)	C8—C9	1.386 (10)
Sn1—O5	2.268 (3)	C9—C10	1.408 (10)
Sn1—N1	2.342 (4)	C9—H9	0.9300
Sn1—C8	2.108 (6)	C10—C11	1.367 (12)
Sn1—C14	2.123 (6)	C10—H10	0.9300
N1—C6	1.335 (6)	C11—C12	1.357 (13)
N1—C2	1.340 (6)	C11—H11	0.9300
O1—C1	1.271 (6)	C12—C13	1.407 (10)
O2—C1	1.233 (6)	C12—H12	0.9300
O3—C7	1.261 (6)	C13—H13	0.9300
O4—C7	1.249 (6)	C14—C15	1.381 (9)
O4—Sn1^{ii}	2.384 (3)	C14—C19	1.394 (8)
O5—H1	0.8347	C15—C16	1.398 (9)
O5—H2	0.9480	C15—H15	0.9300
C1—C2	1.505 (7)	C16—C17	1.363 (10)
C2—C3	1.376 (7)	C16—H16	0.9300
C3—C4	1.368 (8)	C17—C18	1.363 (10)
C3—H3	0.9300	C17—H17	0.9300
C4—C5	1.379 (7)	C18—C19	1.382 (8)
C4—H4	0.9300	C18—H18	0.9300
C5—C6	1.387 (7)	C19—H19	0.9300
C5—H5	0.9300		
O1—Sn1—O3	135.61 (12)	C6—C5—H5	120.8
O1—Sn1—O4^{i}	151.67 (13)	N1—C6—C5	121.0 (4)
O1—Sn1—O5	73.54 (12)	N1—C6—C7	114.5 (4)
O4^{i}—Sn1—O3	72.62 (12)	C5—C6—C7	124.5 (4)
O5—Sn1—O3	150.79 (12)	O4—C7—O3	127.1 (4)
O5—Sn1—O4^{i}	78.37 (12)	O4—C7—C6	116.9 (4)
O1—Sn1—N1	69.74 (13)	O3—C7—C6	116.0 (4)
O5—Sn1—N1	142.76 (13)	C13—C8—C9	119.4 (6)
N1—Sn1—O3	65.98 (12)	C13—C8—Sn1	122.6 (5)
N1—Sn1—O4^{i}	138.57 (12)	C9—C8—Sn1	117.9 (5)
C8—Sn1—O1	95.5 (2)	C8—C9—C10	121.1 (7)
C8—Sn1—O3	87.9 (2)	C8—C9—H9	119.4
C8—Sn1—O4^{i}	86.6 (2)	C10—C9—H9	119.4
C8—Sn1—O5	87.1 (2)	C11—C10—C9	116.5 (9)
C8—Sn1—N1	90.43 (19)	C11—C10—H10	121.7
C8—Sn1—C14	172.6 (2)	C9—C10—H10	121.7
C14—Sn1—O1	91.42 (19)	C12—C11—C10	124.8 (8)
C14—Sn1—O3	88.95 (19)	C12—C11—H11	117.6
C14—Sn1—O4^{i}	86.09 (18)	C10—C11—H11	117.6
C14—Sn1—O5	92.49 (18)	C11—C12—C13	117.7 (8)

C14—Sn1—N1	94.38 (18)	C11—C12—H12	121.2
C6—N1—C2	120.2 (4)	C13—C12—H12	121.2
C6—N1—Sn1	122.6 (3)	C8—C13—C12	120.4 (8)
C2—N1—Sn1	117.1 (3)	C8—C13—H13	119.8
C1—O1—Sn1	122.8 (3)	C12—C13—H13	119.8
C7—O3—Sn1	120.8 (3)	C15—C14—C19	118.4 (6)
C7—O4—Sn1^{ii}	134.1 (3)	C15—C14—Sn1	123.1 (5)
Sn1—O5—H1	120.6	C19—C14—Sn1	118.4 (4)
Sn1—O5—H2	107.0	C14—C15—C16	119.5 (6)
H1—O5—H2	109.4	C14—C15—H15	120.3
O2—C1—O1	126.3 (5)	C16—C15—H15	120.3
O2—C1—C2	117.5 (4)	C17—C16—C15	121.2 (7)
O1—C1—C2	116.2 (4)	C17—C16—H16	119.4
N1—C2—C3	121.5 (4)	C15—C16—H16	119.4
N1—C2—C1	113.8 (4)	C18—C17—C16	119.6 (7)
C3—C2—C1	124.6 (4)	C18—C17—H17	120.2
C4—C3—C2	118.5 (5)	C16—C17—H17	120.2
C4—C3—H3	120.7	C17—C18—C19	120.3 (7)
C2—C3—H3	120.7	C17—C18—H18	119.9
C3—C4—C5	120.4 (5)	C19—C18—H18	119.9
C3—C4—H4	119.8	C18—C19—C14	120.9 (6)
C5—C4—H4	119.8	C18—C19—H19	119.5
C4—C5—C6	118.4 (5)	C14—C19—H19	119.5
C4—C5—H5	120.8		

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H1···O2^{iii}	0.83	1.85	2.671 (3)	167
O5—H2···O3^{i}	0.95	1.74	2.673 (3)	166

Symmetry codes: (iii) −*x*+1, −*y*+1, −*z*; (i) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

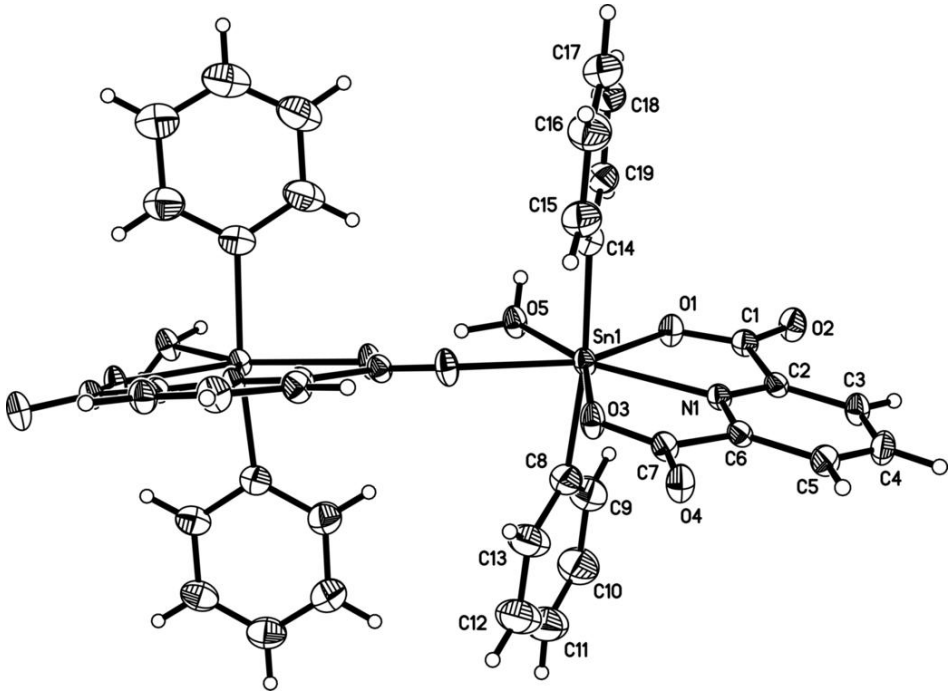
C17
C18
C16
C19
C15
C14
O5
O1
C1
O2
Sn1
C2
C3
O3
N1
C8
C7
C6
C5
C4
C9
O4
C13
C10
C12
C11

Fig. 2