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Key indicators

Single-crystal X-ray study

T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.025

wR factor = 0.059

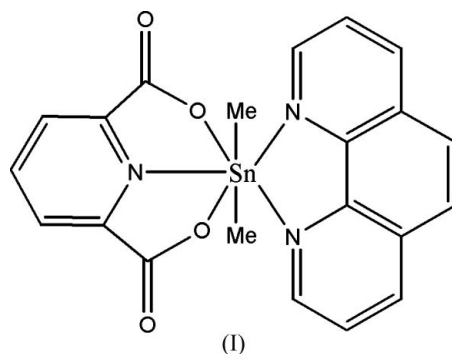
Data-to-parameter ratio = 14.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dimethyl(1,10-phenanthroline)(pyridine-
2,6-dicarboxylato)tin(IV)

The molecule of the title compound, $[\text{Sn}(\text{C}_9\text{H}_3\text{NO}_4)(\text{CH}_3)_2(\text{C}_{12}\text{H}_8\text{N}_2)]$, occupies a special position on a mirror plane, which passes through the Sn, pyridine N and *para*-C, and two methyl C atoms. The Sn atom has a distorted pentagonal-bipyramidal coordination, with the equatorial plane formed by two N atoms of 1,10-phenanthroline [$\text{Sn}-\text{N} = 2.4754(18) \text{ \AA}$], and the pyridine N and two carboxylate O atoms of pyridine-2,6-dicarboxylate [$\text{Sn}-\text{N} = 2.288(2) \text{ \AA}$ and $\text{Sn}-\text{O} = 2.2614(15) \text{ \AA}$]; two methyl groups occupy the axial positions [$\text{Sn}-\text{C} = 2.112(3)$ and $2.096(4) \text{ \AA}$, and $\text{C}-\text{Sn}-\text{C} = 174.26(13)^\circ$].

Comment

Organotin complexes have recently been attracting significant attention due to their wide industrial applications, such as catalysis, organic synthesis, PVC stabilization, pesticides, bactericides, antifouling paints and antiseptic materials, as well as having biological activities (Duboy & Roy, 2003). In order to explore the impact of the structure on the properties of the complexes, and also to analyse the structure-activity relationships, a large number of organotin complexes have been prepared and studied (Gielen, 2002). However, only a few of the synthesized complexes feature mixed ligands, *e.g.* ligands having both N and O coordination sites. Pyridine-2,6-dicarboxylate is one of the most popular ligands of this type, and here we report the structure of the title complex, (I), which contains this ligand.



The molecule of the title complex (Fig. 1) occupies a special position on a mirror plane which passes through atoms Sn1, N2, C1, C2, and C6. Atom Sn1 has a pentagonal-bipyramidal coordination with two phenanthroline N atoms, two carboxylate O atoms and a pyridine N atom in the equatorial plane; two methyl C atoms are in the axial positions. The $\text{Sn}-\text{C}(\text{Me})$, $\text{Sn}-\text{N}$ and $\text{Sn}-\text{O}$ bond lengths involving the pyridine-2,6-dicarboxylate ligand (Table 1) are all close to those

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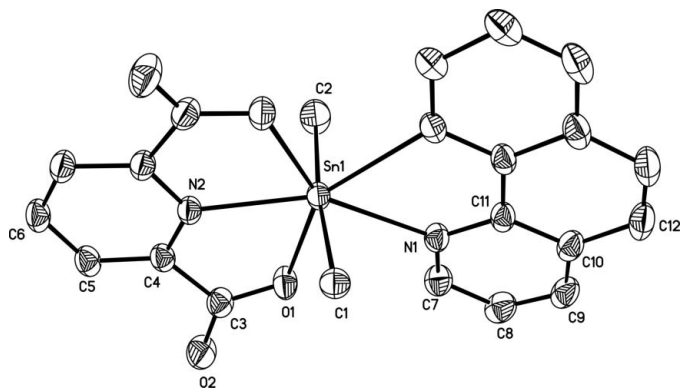


Figure 1

The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted. Unlabelled atoms are related to labelled atoms by the symmetry operator $(x, \frac{1}{2} - y, z)$.

found in bis(dicyclohexylammonium)(oxalato)(pyridine-2,6-dicarboxylato)dibutylstannate(IV) 3.5-hydrate (Ng, 1999) and bis[aquadimethyl(pyridine-2,6-dicarboxylato)tin(IV) hydrate] (Huber *et al.*, 1989). The Sn—N (phenanthroline) bond length is slightly longer than that in bis(3,5-di-*t*-butylcatecholato)tin-1,10-phenanthroline dimethylformamide solvate (Annan *et al.*, 1989). The C3—O1—Sn1 angle is 121.77 (14)°, which is in agreement with the monodentate coordination mode of the carboxylate group (Li *et al.*, 2005). No intermolecular interactions involving the carboxylate C=O group and the Sn atom have been found in the structure of the title compound.

Experimental

All reagents and solvents were used as obtained, without further purification. The reaction was carried out under a nitrogen atmosphere. Pyridine-2,6-dicarboxylic acid (0.167 g, 1 mmol) was added to a solution of sodium ethoxide (0.136 g, 2 mmol) in benzene (20 ml), and the mixture was stirred for 10 min. Dimethyltin dichloride (0.220 g, 1 mmol) and 1,10-phenanthroline (0.198 g, 1 mmol) were then added. The reaction mixture was kept at 313 K for 12 h. After cooling to room temperature, the solution was filtered. The solvent was removed from the filtrate under vacuum and the solid residue was recrystallized from diethyl ether; colourless crystals suitable for X-ray diffraction study were obtained (yield 0.724 g, 85%; m.p. 414 K). Analysis calculated for $C_{21}H_{17}N_3O_4Sn$: C 51.05, H 3.47, N 8.50%; found: C 51.03, H 3.48, N 8.49%.

Crystal data

$[Sn(C_9H_3NO_4)(CH_3)_2(C_{12}H_8N_2)]$
 $M_r = 494.07$
 Monoclinic, $P2_1/m$
 $a = 7.4595$ (8) Å
 $b = 13.754$ (1) Å
 $c = 9.723$ (1) Å
 $\beta = 108.914$ (1)°
 $V = 943.75$ (17) Å³
 $Z = 2$

$D_x = 1.739$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4502 reflections
 $\theta = 2.2$ – 25.2 °
 $\mu = 1.39$ mm⁻¹
 $T = 295$ (2) K
 Plate, colourless
 $0.16 \times 0.14 \times 0.03$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.808$, $T_{max} = 0.959$
 10848 measured reflections

2257 independent reflections
 2099 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.032$
 $\theta_{max} = 27.5$ °
 $h = -9 \rightarrow 9$
 $k = -17 \rightarrow 17$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.059$
 $S = 1.05$
 2257 reflections
 153 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0339P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.46$ e Å⁻³
 $\Delta\rho_{min} = -0.44$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—C2	2.096 (4)	Sn1—N1 ⁱ	2.4754 (18)
Sn1—C1	2.112 (3)	O1—C3	1.274 (3)
Sn1—O1	2.2614 (15)	O2—C3	1.221 (3)
Sn1—N2	2.288 (2)		
C2—Sn1—C1	174.26 (13)	O1 ⁱ —Sn1—N2	69.57 (4)
C2—Sn1—O1	92.17 (6)	O1 ⁱ —Sn1—N1	143.79 (6)
C1—Sn1—O1	89.83 (5)	N1—Sn1—N1 ⁱ	66.83 (9)
O1—Sn1—O1 ⁱ	139.08 (8)		

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

The aromatic H atoms were placed in idealized positions and constrained to ride on their parent atoms with aromatic C—H distances of 0.93 Å. The $U_{iso}(H)$ values were set at $1.2U_{eq}(C)$ of the carrier C atom. The methyl H atoms were located in a difference Fourier map and refined isotropically; their C—H bonds were restrained to 0.96 (3) Å.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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