

Fa-Hui Li, Han-Dong Yin,*
Gang Li and Da-Qi WangCollege of Chemistry and Chemical Engineering,
Liaocheng University, Shandong 252059,
People's Republic of China

Correspondence e-mail: handongyin@163.com

Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$
Disorder in main residue
 R factor = 0.042
 wR factor = 0.117
Data-to-parameter ratio = 16.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Tri-*n*-butyl(5-chloro-6-hydroxypyridine-3-carboxylato)tin(IV)

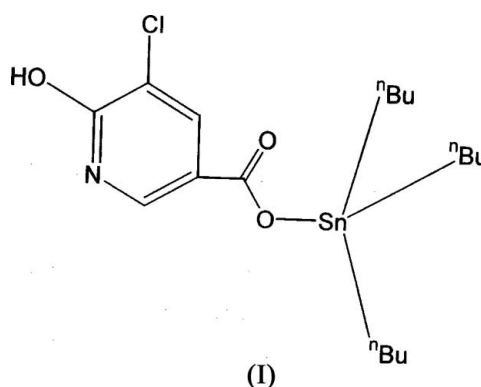
The crystal structure of the title complex, $[\text{Sn}(\text{C}_4\text{H}_9)_3(\text{C}_6\text{H}_3\text{ClNO}_3)]$, possesses an infinite two-dimensional polymeric chain structure in the solid state, resulting from strong intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The Sn atom is five-coordinate with a distorted trigonal bipyramidal geometry.

Received 23 December 2005

Accepted 22 February 2006

Comment

The title complex, (I), consists of three butyl and one 5-chloro-6-hydroxypyridine-3-carboxylate group bonded to the Sn atom (Fig. 1) and has an infinite one-dimensional polymeric chain structure (Fig. 2), due to the $\text{Sn}1\cdots\text{O}3^i$ [symmetry code: (i) $x, y + 1, z$] distance of $2.537(4)\text{ \AA}$, compared to the van der Waals sum of 4.0 \AA (Vollano *et al.*, 1984).



The Sn atom has a distorted trigonal-bipyramidal geometry, with atoms O1 and O3ⁱ in axial positions and C atoms of the three butyl groups in equatorial positions. The distortions away from the ideal trigonal-bipyramidal geometry are not along the Berry coordinate (Hämäläinen & Turpeinen, 1987) and are reflected by the bond angles around the Sn atom (Table 1). The angle O1—Sn1 \cdots O3ⁱ [$178.21(19)^\circ$] deviates from linearity by 1.87° . The sum of the C15—Sn1—C7 [$126.7(3)^\circ$], C15—Sn1—C11 [$115.1(3)^\circ$] and C7—Sn1—C11 [$115.1(3)^\circ$] angles is 356.9° , which shows that these atoms are not coplanar.

The coordination geometry of the Sn atom and the molecular structure of (I) are normal (Xie *et al.*, 1991). The central Sn atom is not coordinated by the N atom of the 5-chloro-6-hydroxynicotinic acid, which is different from the triphenyltin 4-picolinate (Yin *et al.*, 2002).

Strong intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2) between O atoms of the carboxylate groups and H(N) atoms of the pyridine rings result in the formation of two-dimensional polymeric sheets lying in the *bc* plane (Fig. 2).

Experimental

A mixture of tri-*n*-butyltin oxide (596.1 mg, 2.0 mmol) and 5-chloro-6-hydroxynicotinic acid (347.1 mg, 2.0 mmol) in benzene (80 ml) was heated under reflux for 8 h. The resulting clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/methanol (1:1) (yield 712.2 mg, 76%; m.p. 430 K). Analysis calculated for C₁₈H₂₉CINSn: C 46.84, H 6.33, N 3.30%; found: C 46.82, H 6.36, N 3.38%.

Crystal data

[Sn(C₄H₉)₃(C₆H₃CINO₃)]
M_r = 462.57
 Orthorhombic, *P*2₁2₁2₁
a = 8.825 (2) Å
b = 10.823 (3) Å
c = 22.592 (6) Å
V = 2157.9 (10) Å³
Z = 4
D_x = 1.424 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 3993 reflections
 $\theta = 2.5\text{--}26.3^\circ$
 $\mu = 1.32\text{ mm}^{-1}$
T = 298 (2) K
 Block, colorless
 0.38 × 0.24 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.634, *T_{max}* = 0.807
 11314 measured reflections

3815 independent reflections
 2876 reflections with *I* > 2σ(*I*)
R_{int} = 0.055
 $\theta_{\text{max}} = 25.0^\circ$
h = -10 → 10
k = -10 → 12
l = -23 → 26

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.042
wR (*F*²) = 0.117
S = 1.00
 3815 reflections
 226 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 1.3585P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 1.30\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.65\text{ e \AA}^{-3}$
 Absolute structure: Flack (1983), 2193 Friedel pairs
 Flack parameter: -0.05 (5)

Table 1

Selected geometric parameters (Å, °).

Sn1—C15	2.119 (7)	Sn1—O1	2.146 (4)
Sn1—C7	2.133 (6)	Sn1—O3 ⁱ	2.537 (4)
Sn1—C11	2.143 (7)	O3—Sn1 ⁱⁱ	2.537 (4)
C15—Sn1—C7	126.7 (3)	C11—Sn1—O1	92.7 (3)
C15—Sn1—C11	115.1 (3)	C15—Sn1—O3 ⁱ	81.9 (2)
C7—Sn1—C11	115.1 (3)	C7—Sn1—O3 ⁱ	82.4 (2)
C15—Sn1—O1	98.1 (2)	C11—Sn1—O3 ⁱ	88.9 (3)
C7—Sn1—O1	96.2 (2)	O1—Sn1—O3 ⁱ	178.21 (19)

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

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O2 ⁱⁱⁱ	0.86	1.95	2.781 (8)	167

Symmetry code: (iii) -*x*, *y* + ½, -*z* + ¾.

When the crystal structure was solved, atoms C17 and C18 were found to be disordered. During the refinement process with isotropic displacement parameters, the occupancies of the disordered atoms

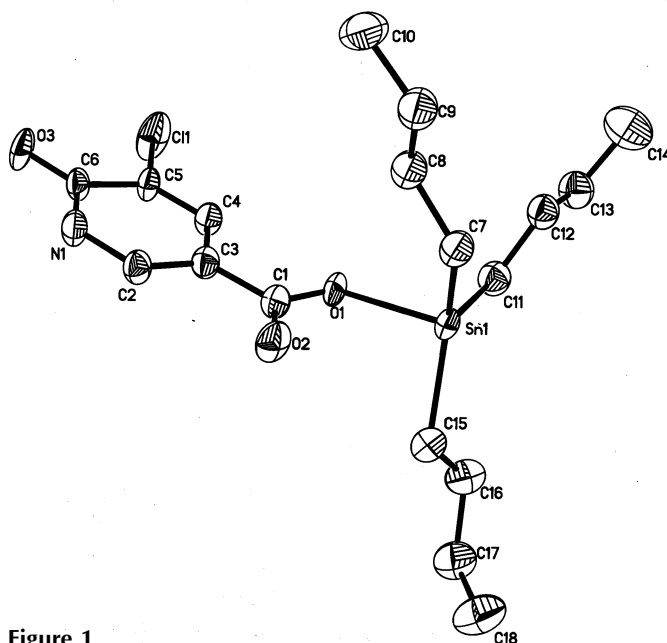


Figure 1

The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms and the minor disorder component have been omitted for clarity.

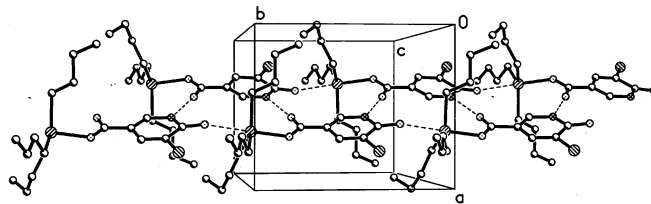


Figure 2

The packing, viewed approximately down the *c* axis. Hydrogen bonds are indicated by dashed lines. H atoms and the minor disorder component have been omitted for clarity.

were refined. These site-occupancy factors were fixed as C17 = 0.80, C17' = 0.20, C18 = 0.80 and C18' = 0.20 during the anisotropic refinement. H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, and constrained to ride on their parent atoms, with *U*_{iso}(H) = *xU*_{eq}(C,N) where *x* = 1.5 for methyl H and *x* = 1.2 for all other H atoms.

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.

We acknowledge financial support from the Shandong Province Science Foundation and the State Key Laboratory of Crystal Material, Shandong University, China.

References

- Bruker (1998). SMART, SAINT and SHELXTL (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). Acta Cryst. A39, 876–881.
- Hämäläinen, R. & Turpeinen, U. (1987). J. Organomet. Chem. 333, 323–327.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Vollano, J. F., Day, R. O. & Holmes, R. R. (1984). *Organometallics*, **3**, 745–750.

Xie, Q. L., Xu, X. H., Wang, H. G., Yao, X. K., Wang, R. J., Zhang, Z. G. & Hu, J. M. (1991). *Acta Chim. Sin.* **49**, 1085–1093.
Yin, H. D., Wang, C. H., Wang, Y., Ma, C. L., Liu, G. F. (2002). *Chin. J. Org. Chem.* **22**, 575–581.