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catena-Poly[[trimethyltin(IV)]- μ -cyclohex-3-ene-1-carboxylato]Yun Ren,^a Rufen Zhang^{b*} and Yang Shi^b

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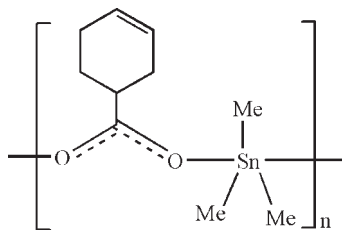
Received 3 December 2009; accepted 17 December 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.013$ Å; R factor = 0.035; wR factor = 0.101; data-to-parameter ratio = 18.6.

The title compound, $[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_9\text{O}_2)]_n$, forms an extended zigzag chain structure propagating parallel to [010]. The Sn atom is in a slightly distorted trigonal-bipyramidal coordination environment with two carboxylate O atoms in the axial and the three methyl groups in equatorial sites. The cyclohexene ring has a distorted half-boat conformation. There is an intramolecular C—H...O hydrogen bond.

Related literature

For related structures, see: Murugavel *et al.* (2001); Ma *et al.* (2006). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

 $[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_9\text{O}_2)]$ $M_r = 288.93$

Monoclinic, $P2_1/n$
 $a = 11.3022$ (15) Å
 $b = 9.8469$ (14) Å
 $c = 12.1468$ (18) Å
 $\beta = 112.148$ (2)°
 $V = 1252.1$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.01$ mm⁻¹
 $T = 298$ K
 $0.45 \times 0.36 \times 0.33$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.464$, $T_{\max} = 0.556$

6095 measured reflections
 2193 independent reflections
 1669 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.101$
 $S = 1.08$
 2193 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C3}-\text{H3B}\cdots\text{O2}$	0.97	2.60	2.926 (10)	100

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: BX2254).

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supporting information

Acta Cryst. (2010). E66, m92 [doi:10.1107/S1600536809054403]

catena-Poly[[trimethyltin(IV)]- μ -cyclohex-3-ene-1-carboxylato]

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S1. Comment

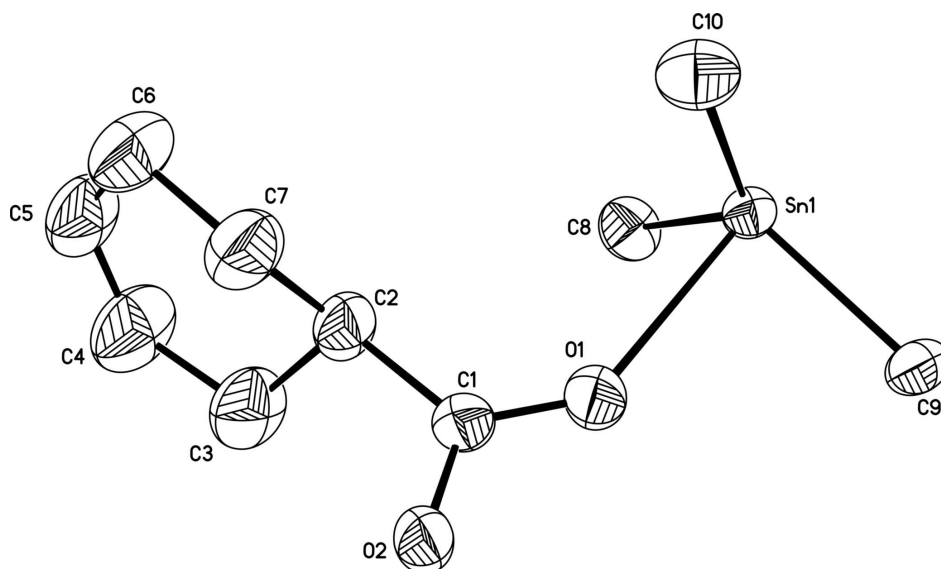
Organotin complexes are attracting more and more attention because of their considerable structural diversity and interesting topologies (Murugavel *et al.* 2001). Herein, we report the crystal structure of the title compound. The title compound, which is shown in Fig.1 forms an infinite zigzag one-dimensional polymeric chain structure. The Sn atom is in a slightly distorted trigonal-bipyramidal coordination environment with two carboxylate O atoms in the axial sites and three methyl groups in equatorial site. The Sn—O, Sn—C bond lengths and the O—Sn \cdots O bond angles are close to the reported organotin compounds (Ma *et al.* 2006). The cyclohexene ring is in a distorted half-boat conformation, the ring-puckering parameters (Cremer & Pople, 1975) are $q_2 = 0.380(1) \text{ \AA}$, $q_3 = -0.314(1) \text{ \AA}$, $Q = 0.492(9)^\circ$ and $\phi_2 = 165.1(2)^\circ$. There is an intramolecular C—H \cdots O hydrogen bond (H3B—O2 2.60, C3—O2 2.926(10) \AA , C3—H3B \cdots O2 100 $^\circ$).

S2. Experimental

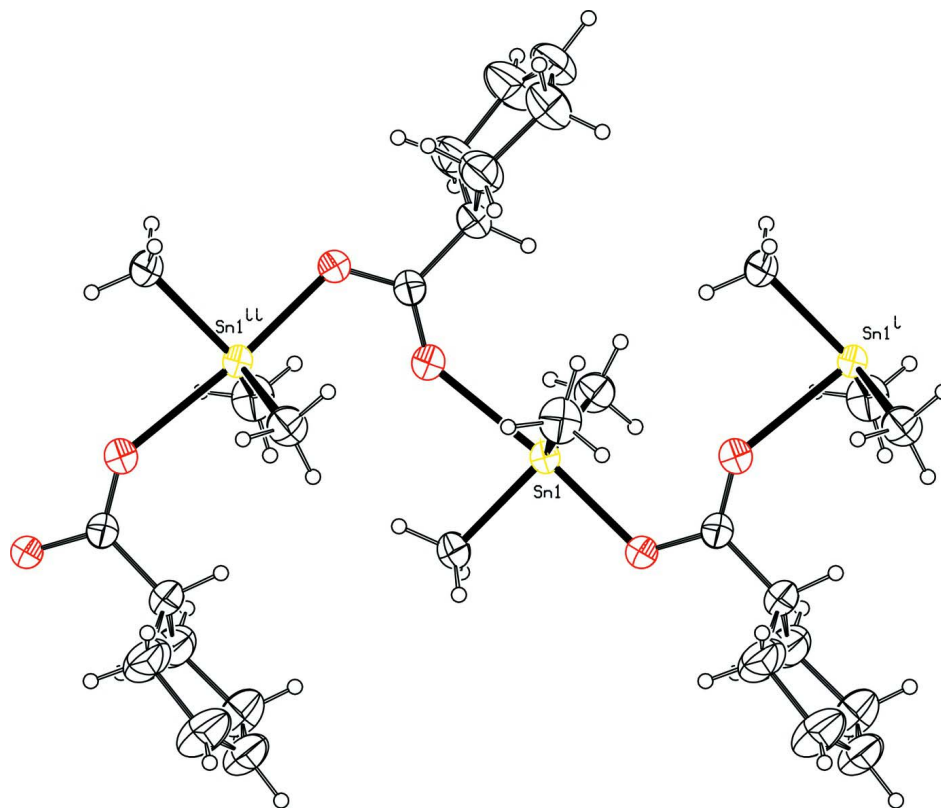
The reaction was carried out under nitrogen atmosphere. 3-cyclohexene-1-carboxylic acid (1 mmol) and sodium ethoxide (1 mmol) were added to a stirred solution of benzene (30 ml) in a Schlenk flask and stirred for 0.5 h. trimethyltin chloride (1 mmol) was then added to the reactor and the reaction mixture was stirred for 12 h at room temperature. The resulting clear solution was evaporated under vacuum. The product was crystallized from ether to yield colorless blocks of the title compound (yield 86% m.p.448 K). Anal. Calcd (%) for $C_{10}H_{18}O_2Sn_1$ (Mr = 288.93): C, 41.57; H, 6.28; Found (%): C, 41.76; H, 6.01.

S3. Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93–0.98 \AA , and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H and $1.2U_{eq}(C)$, for the others.

**Figure 1**

Part of the polymeric structure and the atom-numbering scheme of the title compound. Displacement ellipsoids are shown at the 30% probability level and H atoms have been omitted for clarity.

**Figure 2**

Part of the structure of (I) showing the one-dimensional chain along the b axis [symmetry codes: (i) $1/2-x, -1/2+y, 1/2-z$; (ii) $1/2-x, 1/2+y, 1/2-z$].

catena-Poly[[trimethyltin(IV)]- μ -cyclohex-3-ene-1-carboxylato]

Crystal data

[Sn(CH₃)₃(C₇H₉O₂)]
M_r = 288.93
 Monoclinic, *P*2₁/*n*
 Hall symbol: -*P* 2₁
a = 11.3022 (15) Å
b = 9.8469 (14) Å
c = 12.1468 (18) Å
 β = 112.148 (2)°
V = 1252.1 (3) Å³
Z = 4

F(000) = 576
D_x = 1.533 Mg m⁻³
 Mo *K* α radiation, λ = 0.71073 Å
 Cell parameters from 2901 reflections
 θ = 2.8–25.7°
 μ = 2.01 mm⁻¹
T = 298 K
 Block, colourless
 0.45 × 0.36 × 0.33 mm

Data collection

Siemens SMART CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
T_{min} = 0.464, *T_{max}* = 0.556

6095 measured reflections
 2193 independent reflections
 1669 reflections with *I* > 2 σ (*I*)
R_{int} = 0.033
 θ_{\max} = 25.0°, θ_{\min} = 2.1°
h = -13→13
k = -10→11
l = -14→9

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2 σ (*F*²)] = 0.035
wR(*F*²) = 0.101
S = 1.08
 2193 reflections
 118 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
 $w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 2.3141P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.53 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.49 \text{ e \AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> */ <i>U_{eq}</i>
Sn1	0.17568 (3)	0.09824 (4)	0.23917 (3)	0.04584 (17)
O1	0.3014 (4)	0.2882 (4)	0.2205 (5)	0.0699 (12)
O2	0.4496 (4)	0.4411 (4)	0.2415 (4)	0.0652 (12)
C1	0.4091 (6)	0.3183 (6)	0.2231 (6)	0.0574 (15)
C2	0.4961 (6)	0.2138 (7)	0.2052 (7)	0.0694 (18)
H2	0.4529	0.1255	0.1915	0.083*
C3	0.5310 (8)	0.2458 (9)	0.1026 (8)	0.101 (3)
H3A	0.4539	0.2451	0.0313	0.121*
H3B	0.5654	0.3373	0.1125	0.121*
C4	0.6246 (10)	0.1532 (11)	0.0847 (10)	0.118 (3)
H4	0.6277	0.1437	0.0097	0.142*
C5	0.7028 (9)	0.0848 (9)	0.1741 (10)	0.098 (3)
H5	0.7612	0.0275	0.1603	0.118*

C6	0.7047 (9)	0.0922 (9)	0.2906 (10)	0.104 (3)
H6A	0.7920	0.1077	0.3449	0.125*
H6B	0.6781	0.0051	0.3106	0.125*
C7	0.6205 (7)	0.2020 (9)	0.3105 (8)	0.091 (2)
H7A	0.6032	0.1806	0.3809	0.109*
H7B	0.6650	0.2883	0.3236	0.109*
C8	0.1852 (7)	0.0177 (7)	0.0817 (6)	0.0684 (18)
H8A	0.2669	-0.0244	0.0994	0.103*
H8B	0.1742	0.0896	0.0253	0.103*
H8C	0.1189	-0.0486	0.0489	0.103*
C9	0.0327 (6)	0.2468 (6)	0.2198 (7)	0.072 (2)
H9A	0.0032	0.2384	0.2840	0.109*
H9B	-0.0375	0.2333	0.1456	0.109*
H9C	0.0679	0.3358	0.2212	0.109*
C10	0.3183 (7)	0.0735 (7)	0.4101 (6)	0.075 (2)
H10A	0.3965	0.0455	0.4031	0.113*
H10B	0.2917	0.0057	0.4528	0.113*
H10C	0.3315	0.1581	0.4523	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0474 (3)	0.0394 (2)	0.0517 (3)	0.00229 (17)	0.01983 (19)	0.00063 (18)
O1	0.065 (3)	0.049 (2)	0.105 (4)	-0.003 (2)	0.043 (3)	-0.001 (3)
O2	0.063 (3)	0.046 (2)	0.102 (4)	-0.0029 (19)	0.048 (3)	-0.007 (2)
C1	0.063 (4)	0.046 (3)	0.070 (4)	0.004 (3)	0.032 (3)	0.003 (3)
C2	0.066 (4)	0.053 (4)	0.098 (5)	0.005 (3)	0.041 (4)	-0.009 (4)
C3	0.114 (6)	0.101 (6)	0.110 (7)	0.027 (5)	0.068 (6)	-0.007 (5)
C4	0.129 (8)	0.132 (8)	0.110 (8)	0.048 (7)	0.065 (7)	-0.001 (7)
C5	0.090 (6)	0.092 (6)	0.123 (8)	0.026 (5)	0.054 (6)	-0.011 (6)
C6	0.097 (6)	0.092 (6)	0.121 (8)	0.033 (5)	0.039 (6)	0.008 (6)
C7	0.085 (5)	0.087 (6)	0.103 (6)	0.021 (5)	0.040 (5)	0.005 (5)
C8	0.077 (4)	0.073 (4)	0.059 (4)	-0.006 (4)	0.030 (4)	-0.006 (4)
C9	0.061 (4)	0.049 (3)	0.111 (6)	0.012 (3)	0.036 (4)	0.016 (4)
C10	0.079 (5)	0.080 (5)	0.054 (4)	0.002 (4)	0.009 (4)	0.006 (4)

Geometric parameters (Å, °)

Sn1—C10	2.108 (7)	C5—C6	1.408 (14)
Sn1—C8	2.110 (6)	C5—H5	0.9300
Sn1—C9	2.126 (6)	C6—C7	1.519 (11)
Sn1—O2 ⁱ	2.169 (4)	C6—H6A	0.9700
Sn1—O1	2.411 (4)	C6—H6B	0.9700
O1—C1	1.241 (7)	C7—H7A	0.9700
O2—C1	1.282 (7)	C7—H7B	0.9700
O2—Sn1 ⁱⁱ	2.169 (4)	C8—H8A	0.9600
C1—C2	1.495 (8)	C8—H8B	0.9600
C2—C3	1.475 (10)	C8—H8C	0.9600

C2—C7	1.506 (10)	C9—H9A	0.9600
C2—H2	0.9800	C9—H9B	0.9600
C3—C4	1.474 (11)	C9—H9C	0.9600
C3—H3A	0.9700	C10—H10A	0.9600
C3—H3B	0.9700	C10—H10B	0.9600
C4—C5	1.300 (13)	C10—H10C	0.9600
C4—H4	0.9300		
C10—Sn1—C8	124.6 (3)	C6—C5—H5	118.0
C10—Sn1—C9	117.1 (3)	C5—C6—C7	115.0 (8)
C8—Sn1—C9	117.0 (3)	C5—C6—H6A	108.5
C10—Sn1—O2 ⁱ	95.7 (2)	C7—C6—H6A	108.5
C8—Sn1—O2 ⁱ	95.1 (2)	C5—C6—H6B	108.5
C9—Sn1—O2 ⁱ	90.2 (2)	C7—C6—H6B	108.5
C10—Sn1—O1	85.6 (2)	H6A—C6—H6B	107.5
C8—Sn1—O1	88.4 (2)	C2—C7—C6	111.1 (7)
C9—Sn1—O1	84.6 (2)	C2—C7—H7A	109.4
O2 ⁱ —Sn1—O1	174.62 (14)	C6—C7—H7A	109.4
C1—O1—Sn1	142.2 (4)	C2—C7—H7B	109.4
C1—O2—Sn1 ⁱⁱ	119.1 (4)	C6—C7—H7B	109.4
O1—C1—O2	120.8 (5)	H7A—C7—H7B	108.0
O1—C1—C2	121.6 (6)	Sn1—C8—H8A	109.5
O2—C1—C2	117.6 (5)	Sn1—C8—H8B	109.5
C3—C2—C1	112.1 (6)	H8A—C8—H8B	109.5
C3—C2—C7	105.8 (6)	Sn1—C8—H8C	109.5
C1—C2—C7	112.6 (6)	H8A—C8—H8C	109.5
C3—C2—H2	108.8	H8B—C8—H8C	109.5
C1—C2—H2	108.8	Sn1—C9—H9A	109.5
C7—C2—H2	108.8	Sn1—C9—H9B	109.5
C4—C3—C2	115.3 (8)	H9A—C9—H9B	109.5
C4—C3—H3A	108.4	Sn1—C9—H9C	109.5
C2—C3—H3A	108.4	H9A—C9—H9C	109.5
C4—C3—H3B	108.4	H9B—C9—H9C	109.5
C2—C3—H3B	108.4	Sn1—C10—H10A	109.5
H3A—C3—H3B	107.5	Sn1—C10—H10B	109.5
C5—C4—C3	119.8 (9)	H10A—C10—H10B	109.5
C5—C4—H4	120.1	Sn1—C10—H10C	109.5
C3—C4—H4	120.1	H10A—C10—H10C	109.5
C4—C5—C6	124.1 (8)	H10B—C10—H10C	109.5
C4—C5—H5	118.0		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3B \cdots O2	0.97	2.60	2.926 (10)	100