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catena-Poly[[trimethyltin(IV)]-μ-2-[(3-oxocyclohex-1-enyl)amino]-benzoato-κ^2O:O']

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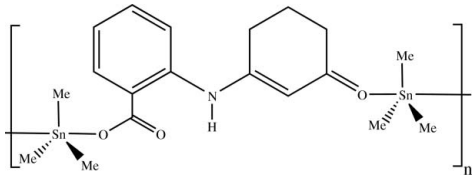
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.007 Å; R factor = 0.024; wR factor = 0.087; data-to-parameter ratio = 12.1.

The Sn atom in the title compound, $[Sn(CH_3)_3(C_{13}H_{13}NO_3)]_n$, is five-coordinate in a trigonal–bipyramidal environment. An intramolecular N—H···O hydrogen bond is present.

Related literature

For related literature, see: Vieira *et al.* (2007).



Experimental

Crystal data

$[Sn(CH_3)_3(C_{13}H_{13}NO_3)]$
M_r = 394.03
Orthorhombic, *Fdd*2
a = 30.626 (5) Å
b = 12.981 (2) Å
c = 17.164 (2) Å
V = 6823.6 (19) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 1.51 mm^{-1}
T = 273 (2) K
0.2 × 0.2 × 0.2 mm

Data collection

Siemens *P*4 diffractometer
Absorption correction: none
7294 measured reflections
2329 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
3 standard reflections
every 97 reflections
intensity decay: 4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.087
S = 0.74
2329 reflections
193 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Absolute structure: Flack (1983), with 1560 Friedel pairs
Flack parameter: −0.04 (4)

Table 1
Selected geometric parameters (Å, °).

Sn—C1	2.112 (5)	Sn—O3	2.496 (4)
Sn—C2	2.111 (6)	C10—N	1.396 (7)
Sn—C3	2.113 (6)	N—C11^{i}	1.376 (6)
Sn—O1	2.174 (4)		
C1—Sn—C2	120.1 (3)	C1—Sn—O3	79.46 (19)
C1—Sn—C3	125.5 (3)	C2—Sn—O3	91.4 (2)
C2—Sn—C3	112.5 (3)	C3—Sn—O3	86.3 (2)
C1—Sn—O1	94.73 (19)	O1—Sn—O3	174.13 (13)
C2—Sn—O1	90.8 (2)	C11^{i}—N—C10	128.5 (4)
C3—Sn—O1	97.9 (2)		

Symmetry code: (i) $-x+\frac{3}{4}, y+\frac{3}{4}, z+\frac{1}{4}$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H1N···O2	0.91 (6)	1.96 (6)	2.631 (5)	129 (5)

Data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2023).

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supplementary materials

Acta Cryst. (2007). E**63**, m2587 [**doi:10.1107/S1600536807044273**]

catena-Poly[[trimethyltin(IV)]-μ-2-[(3-oxocyclohex-1-enyl)amino]benzoato-κ^2O:O']

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Comment

The reaction of 2-(3-oxocyclohex-1-enyl)benzoic acid (Vieira *et al.*, 2007) with $Sn(CH_3)_3Cl$ in the presence of triethylamine yielded the title complex. The X-ray crystallographic study revealed that it crystallizes forming an infinity double-polymeric chain structure, where the anionic ligand bridges two tin centre *via* the monodentate carboxylic moiety and the C═O fragment, Fig. 1. The structure possess one tin atom surrounded by three methyl groups and two oxygen atoms, describing an almost perfect trigonal bypiramid. The equatorial corners are occupied by the methyl groups and the axial positions by the oxygen atoms. The angles C1—Sn—C2 and O1—Sn—O3 are all near 120° and 180° as expected for a trigonal bypiramid.

Experimental

To a round-botton flask charged with 3-[(carboxyphenyl)amino]cyclohexen-2-one (1.0 g, 4.32 mmol) and triethylamine (0.6 ml, 4.32 mmol) dissolved in methanol (20 ml), was added trimethyltin chloride (0.86 g, 4.32 mmol). The X-ray quality crystals were obtained from a methanol/water (3:1) solution. IR (ν/cm^{-1}): 473 ($\nu_{Sn—O}$). ^{1}H NMR (δ, $CDCl_3$): 8.63 d (C6), 8.0 m (C8, C9), 7.7 m (C7) 6.23 s (C16), 3.0 m (C14), 2.8 m (C12), 2.47 m (C13), 1.58 s (C1, 2, 3). ^{13}C-NMR (δ, $CDCl_3$): 199.5 (C15), 173.4 (C4), 162.5 (C11), 141.8 (C10), 133.9 (C6), 133.6 (C8), 124.3 (C5), 124.2 (C2), 101.58 (C7), 101.56 (C16), 37.5 (C14), 31.2 (C12), 22.8 (C13), 11.7 (C1, 2, 3), ^{119}Sn-NMR (δ, $CDCl_3$): 97.6, ^{119}Sn Mossbauer (mm.s^{-1}): δ 1.32, Δ 3.52, Elemental Analysis (%) for $C_{16}H_{21}NO_3Sn$ found (calc.): C 48.41 (48.76), H 5.37 (5.38), N 3.46 (3.55).

Refinement

Fourier difference in the structure determination stage evidenced most of the H atoms in the structure; nevertheless, their positions were subsequently calculated and refined using a riding model approximation. All non-H atoms were refined anisotropically.

Figures

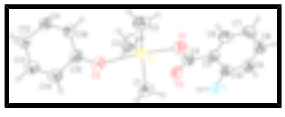

Fig. 1. Monomeric structure of poly-[trimethyltin-3-{(carboxyphenyl)amino} cyclohexen-2-one]

<it>*catena*</it>-Poly[[trimethyltin(IV)]-μ-2-[(3-oxocyclohex-1- enyl)amino]benzoato-κ^2<it>O</it>:<it>O</it>^{1}]

Crystal data

$[Sn(CH_3)_3(C_{13}H_{13}NO_3)]$	D_x = 1.534 Mg m^{-3}
M_r = 394.03	Melting point = 440–443 K
Orthorhombic, *Fdd*2	Mo *K*α radiation λ = 0.71073 Å
a = 30.626 (5) Å	Cell parameters from 26 reflections
b = 12.981 (2) Å	θ = 11.8–12.5º
c = 17.164 (2) Å	μ = 1.51 mm^{-1}
V = 6823.6 (19) Å^3	*T* = 273 (2) K
Z = 16	Prismatic, colourless
F_{000} = 3168	0.2 × 0.2 × 0.2 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.039
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.1º
T = 273(2) K	*h* = −36→36
2θ/ω scans	*k* = −15→11
Absorption correction: none	*l* = −19→12
7294 measured reflections	3 standard reflections
2329 independent reflections	every 97 reflections
2099 reflections with $I > 2\sigma(I)$	intensity decay: 4%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.024	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.087	$(\Delta/\sigma)_{max} < 0.001$
S = 0.74	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
2329 reflections	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
193 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with how many Friedel pairs?
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.04 (4)
Secondary atom site location: difference Fourier map	

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}
Sn	0.411524 (8)	−0.002008 (19)	0.26150 (5)	0.05087 (14)
C1	0.43775 (19)	0.1345 (4)	0.2137 (4)	0.0718 (15)
H1A	0.4312	0.1915	0.2474	0.108*
H1B	0.4251	0.1466	0.1634	0.108*
H1C	0.4688	0.1276	0.2087	0.108*
C2	0.4509 (2)	−0.0989 (5)	0.3309 (4)	0.0772 (16)
H2A	0.4442	−0.0880	0.3848	0.116*
H2B	0.4811	−0.0832	0.3218	0.116*
H2C	0.4454	−0.1695	0.3175	0.116*
C3	0.3535 (2)	−0.0753 (5)	0.2245 (4)	0.089 (2)
H3A	0.3308	−0.0634	0.2622	0.134*
H3B	0.3585	−0.1480	0.2196	0.134*
H3C	0.3447	−0.0477	0.1751	0.134*
O1	0.38557 (11)	0.0726 (2)	0.3650 (2)	0.0602 (8)
O2	0.33660 (11)	0.1547 (2)	0.2914 (2)	0.0616 (9)
O3	0.44514 (15)	−0.0706 (3)	0.1396 (2)	0.0771 (11)
C4	0.35365 (15)	0.1367 (3)	0.3546 (3)	0.0513 (10)
C5	0.33861 (13)	0.1881 (3)	0.4283 (3)	0.0444 (10)
C6	0.35136 (15)	0.1499 (3)	0.5001 (3)	0.0558 (11)
H6A	0.3703	0.0939	0.5021	0.067*
C7	0.33636 (18)	0.1936 (5)	0.5696 (3)	0.0673 (15)
H7A	0.3450	0.1670	0.6176	0.081*
C8	0.3084 (2)	0.2772 (5)	0.5656 (3)	0.0643 (15)
H8A	0.2977	0.3059	0.6115	0.077*
C9	0.29629 (16)	0.3184 (4)	0.4959 (3)	0.0561 (11)
H9A	0.2778	0.3753	0.4950	0.067*
C10	0.31093 (15)	0.2770 (4)	0.4268 (3)	0.0474 (10)
N	0.29828 (13)	0.3157 (3)	0.3544 (3)	0.0510 (9)
H1N	0.3021 (18)	0.279 (5)	0.310 (3)	0.061*
C11	0.45673 (14)	−0.3331 (3)	0.0821 (3)	0.0449 (10)
C12	0.47411 (17)	−0.3229 (4)	0.0014 (3)	0.0594 (12)
H12A	0.4622	−0.3776	−0.0306	0.071*
H12B	0.5056	−0.3310	0.0026	0.071*

C13	0.4631 (2)	−0.2201 (4)	−0.0352 (3)	0.0703 (15)
H13A	0.4799	−0.2115	−0.0827	0.084*
H13B	0.4323	−0.2187	−0.0488	0.084*
C14	0.47305 (19)	−0.1322 (4)	0.0200 (4)	0.0665 (14)
H14A	0.4616	−0.0688	−0.0018	0.080*
H14B	0.5045	−0.1247	0.0245	0.080*
C15	0.45398 (15)	−0.1476 (3)	0.1002 (3)	0.0528 (11)
C16	0.44788 (16)	−0.2510 (3)	0.1278 (3)	0.0490 (11)
H16A	0.4376	−0.2617	0.1782	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn	0.05684 (19)	0.03705 (18)	0.0587 (2)	0.00106 (12)	−0.00554 (19)	0.00251 (11)
C1	0.098 (4)	0.039 (2)	0.079 (4)	0.003 (2)	0.014 (3)	0.005 (2)
C2	0.094 (4)	0.064 (3)	0.073 (4)	0.011 (3)	0.001 (3)	0.020 (3)
C3	0.084 (4)	0.072 (4)	0.112 (6)	−0.009 (3)	−0.016 (4)	−0.026 (4)
O1	0.071 (2)	0.0529 (19)	0.057 (2)	0.0084 (15)	−0.0044 (17)	0.0016 (15)
O2	0.091 (3)	0.0457 (16)	0.049 (2)	0.0143 (15)	−0.0092 (17)	−0.0075 (14)
O3	0.117 (3)	0.0449 (18)	0.069 (2)	0.0005 (19)	0.007 (2)	−0.0108 (19)
C4	0.068 (3)	0.042 (2)	0.044 (3)	−0.012 (2)	0.001 (2)	0.001 (2)
C5	0.051 (3)	0.038 (2)	0.045 (2)	−0.0123 (16)	−0.0020 (18)	−0.005 (2)
C6	0.059 (2)	0.049 (2)	0.059 (3)	−0.0073 (18)	−0.010 (2)	0.011 (2)
C7	0.087 (4)	0.072 (4)	0.042 (3)	−0.023 (3)	−0.006 (2)	0.016 (3)
C8	0.083 (4)	0.063 (3)	0.047 (3)	−0.020 (3)	0.019 (3)	−0.006 (3)
C9	0.066 (3)	0.048 (2)	0.054 (3)	−0.009 (2)	0.002 (2)	−0.009 (2)
C10	0.049 (2)	0.043 (2)	0.049 (3)	−0.0119 (18)	0.004 (2)	−0.005 (2)
N	0.067 (2)	0.0390 (19)	0.047 (2)	−0.0001 (16)	−0.0032 (18)	−0.0048 (17)
C11	0.047 (2)	0.038 (2)	0.049 (3)	−0.0034 (17)	0.0002 (18)	−0.0043 (19)
C12	0.077 (3)	0.049 (2)	0.053 (3)	0.000 (2)	0.016 (3)	0.003 (3)
C13	0.102 (4)	0.049 (3)	0.061 (4)	−0.003 (3)	0.007 (3)	0.006 (3)
C14	0.089 (4)	0.047 (2)	0.064 (4)	−0.008 (2)	0.009 (3)	0.002 (3)
C15	0.059 (3)	0.038 (2)	0.061 (3)	−0.0014 (18)	0.004 (2)	−0.008 (2)
C16	0.054 (3)	0.043 (2)	0.050 (3)	0.0021 (16)	0.004 (2)	−0.0017 (17)

Geometric parameters (Å, °)

Sn—C1	2.112 (5)	C7—H7A	0.9300
Sn—C2	2.111 (6)	C8—C9	1.362 (8)
Sn—C3	2.113 (6)	C8—H8A	0.9300
Sn—O1	2.174 (4)	C9—C10	1.377 (7)
Sn—O3	2.496 (4)	C9—H9A	0.9300
C1—H1A	0.9600	C10—N	1.396 (7)
C1—H1B	0.9600	N—C11^{i}	1.376 (6)
C1—H1C	0.9600	N—H1N	0.91 (6)
C2—H2A	0.9600	C11—C16	1.351 (6)
C2—H2B	0.9600	C11—N^{ii}	1.376 (6)
C2—H2C	0.9600	C11—C12	1.489 (7)

C3—H3A	0.9600	C12—C13	1.513 (8)
C3—H3B	0.9600	C12—H12A	0.9700
C3—H3C	0.9600	C12—H12B	0.9700
O1—C4	1.296 (5)	C13—C14	1.515 (8)
O2—C4	1.227 (6)	C13—H13A	0.9700
O3—C15	1.237 (6)	C13—H13B	0.9700
C4—C5	1.503 (7)	C14—C15	1.508 (8)
C5—C6	1.384 (7)	C14—H14A	0.9700
C5—C10	1.432 (7)	C14—H14B	0.9700
C6—C7	1.400 (8)	C15—C16	1.436 (6)
C6—H6A	0.9300	C16—H16A	0.9300
C7—C8	1.384 (9)		
C1—Sn—C2	120.1 (3)	C6—C7—H7A	120.7
C1—Sn—C3	125.5 (3)	C9—C8—C7	121.3 (5)
C2—Sn—C3	112.5 (3)	C9—C8—H8A	119.3
C1—Sn—O1	94.73 (19)	C7—C8—H8A	119.3
C2—Sn—O1	90.8 (2)	C8—C9—C10	121.0 (5)
C3—Sn—O1	97.9 (2)	C8—C9—H9A	119.5
C1—Sn—O3	79.46 (19)	C10—C9—H9A	119.5
C2—Sn—O3	91.4 (2)	C9—C10—N	122.4 (5)
C3—Sn—O3	86.3 (2)	C9—C10—C5	119.5 (5)
O1—Sn—O3	174.13 (13)	N—C10—C5	118.1 (4)
Sn—C1—H1A	109.5	$C11^{i}$—N—C10	128.5 (4)
Sn—C1—H1B	109.5	$C11^{i}$—N—H1N	106 (4)
H1A—C1—H1B	109.5	C10—N—H1N	122 (4)
Sn—C1—H1C	109.5	C16—C11—N^{ii}	124.6 (4)
H1A—C1—H1C	109.5	C16—C11—C12	122.8 (4)
H1B—C1—H1C	109.5	N^{ii}—C11—C12	112.6 (4)
Sn—C2—H2A	109.5	C11—C12—C13	112.7 (4)
Sn—C2—H2B	109.5	C11—C12—H12A	109.1
H2A—C2—H2B	109.5	C13—C12—H12A	109.1
Sn—C2—H2C	109.5	C11—C12—H12B	109.1
H2A—C2—H2C	109.5	C13—C12—H12B	109.1
H2B—C2—H2C	109.5	H12A—C12—H12B	107.8
Sn—C3—H3A	109.5	C14—C13—C12	111.0 (5)
Sn—C3—H3B	109.5	C14—C13—H13A	109.4
H3A—C3—H3B	109.5	C12—C13—H13A	109.4
Sn—C3—H3C	109.5	C14—C13—H13B	109.4
H3A—C3—H3C	109.5	C12—C13—H13B	109.4
H3B—C3—H3C	109.5	H13A—C13—H13B	108.0
C4—O1—Sn	116.7 (3)	C13—C14—C15	113.1 (4)
C15—O3—Sn	146.8 (4)	C13—C14—H14A	109.0
O2—C4—O1	124.4 (4)	C15—C14—H14A	109.0
O2—C4—C5	122.0 (4)	C13—C14—H14B	109.0
O1—C4—C5	113.6 (4)	C15—C14—H14B	109.0
C6—C5—C10	118.1 (4)	H14A—C14—H14B	107.8
C6—C5—C4	120.2 (4)	O3—C15—C16	123.1 (5)
C10—C5—C4	121.6 (4)	O3—C15—C14	118.5 (4)

C5—C6—C7	121.4 (4)	C16—C15—C14	118.4 (4)
C5—C6—H6A	119.3	C11—C16—C15	121.3 (4)
C7—C6—H6A	119.3	C11—C16—H16A	119.4
C8—C7—C6	118.6 (5)	C15—C16—H16A	119.4
C8—C7—H7A	120.7		

Symmetry codes: (i) −*x*+3/4, *y*+3/4, *z*+1/4; (ii) −*x*+3/4, *y*−3/4, *z*−1/4.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1N···O2	0.91 (6)	1.96 (6)	2.631 (5)	129 (5)

Fig. 1