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Di-*n*-butyldicinnamatotin(IV)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.016 Å
R factor = 0.044
wR factor = 0.138
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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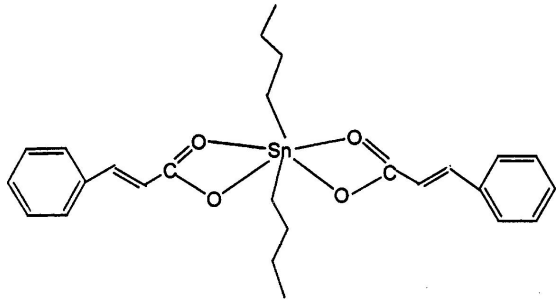
In the title complex, $[Sn(C_4H_9)_2(C_9H_7O_2)_2]$, the central Sn^{IV} atom, lying on a twofold rotation axis, is situated in a skew-trapezoidal bipyramidal geometry and the basal plane is defined by two asymmetrically chelating carboxylate groups. The two remaining positions are occupied by two *n*-butyl groups which lie over the weaker Sn—O bonds.

Comment

From Fig. 1 it can be seen that the Sn atom in the title compound, (I), exists in a skew-trapezoidal planar geometry in which the basal plane is defined by the four O atoms of the two chelating carboxylate groups of the cinnamate ligands. The two remaining positions are occupied by two *n*-butyl groups which lie over the weaker Sn—O bonds. The Sn atom lies on a twofold rotation axis.



(I)

The coordination geometry of the central Sn^{IV} atom can be described as distorted octahedral, with atoms C10, C10^{i}, O1 and O1^{i} occupying the equatorial positions [symmetry code: (i) $-x+2, -y+2, z$], and O2 and O2^{i} occupying the axial positions. The molecular structure consists of a monomer with a hexacoordinated Sn^{IV} atom surrounded by four O atoms and two *n*-butyl groups. The coordination polyhedron of the Sn^{IV} atom can also be described as a highly distorted octahedron, as a bicapped tetrahedron with O atoms, O2 and O2^{i}, capping two faces, or as a skewed trapezoidal bipyramid with the butyl groups in apical positions. The carboxylates are bidentate, with one pair of short Sn—O bonds [Sn1—O1 and Sn1—O1^{i} of 2.225 (6) Å] and one pair of weaker Sn—O bonds [Sn1—O2 and Sn1—O2^{i} of 2.547 (5) Å]. Both carboxylate groups are nearly coplanar. The structure of (I) is similar to those reviewed by Tiekink (1991) and to that of di-*n*-butyltin bis(*o*-aminobenzoate) (Meriem *et al.*, 1990). There is a good agreement in their structural parameters: the short Sn—O distances lie in the range 2.077–2.156 Å and the long Sn—O distances in the range 2.510–2.711 Å. The angles lie in the range 79.5–84.4° for O1—Sn1—O1^{i} [78.7 (3)° in the title

compound], 165.3–172.5° for O2—Sn1—O2^{i} [173.0 (3)°] and 130.6–146.2° for C10—Sn1—C10^{i} [135.2 (6)°]. All these structures display either an approximate or crystallographically imposed C_2 symmetry, with the exception of $(n\text{-}C_4H_9)_2Sn(O_2CC_6H_4NH_2)_2$ (Meriem *et al.*, 1990), in which the amino groups of the ligands are hydrogen bonded to carboxylate O atoms.

Experimental

To a benzene suspension of $(n\text{-Bu})_2SnO$ (2 mmol) was added a benzene solution of cinnamic acid (4 mmol) and the mixture was refluxed for 5 h, with water formed during the reaction being removed azeotropically with a Dean–Stark apparatus. The clear solution obtained after filtration was evaporated in a vacuum to give a white solid. The product was recrystallized from benzene–ether and colorless crystals suitable for X-ray diffraction were obtained (m.p. 445 K). Analysis calculated for $C_{26}H_{32}O_4Sn$: C 59.18, H 6.07%; found: C 59.22, H 6.01%.

Crystal data

$[Sn(C_4H_9)_2(C_9H_7O_2)_2]$
M_r = 527.21
Orthorhombic, *Pba*2
a = 9.833 (3) Å
b = 25.403 (4) Å
c = 5.1578 (19) Å
V = 1288.3 (6) Å^3
Z = 2
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2295 reflections
θ = 2.2–21.7°
μ = 1.02 mm^{-1}
T = 298 (2) K
Block, colorless
0.47 × 0.38 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.646, T_{max} = 0.807
6259 measured reflections
2247 independent reflections
1536 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 25.0°
$h = -11 \rightarrow 10$
$k = -24 \rightarrow 30$
$l = -6 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.138
S = 1.00
2247 reflections
141 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: Flack (1983), 972 Friedel reflections
Flack parameter = 0.11 (10)

Table 1
Selected geometric parameters (Å, °).

Sn1—C10	2.099 (7)	Sn1—O2	2.547 (5)
Sn1—O1	2.115 (6)		
C10—Sn1—C10^{i}	135.2 (6)	C10^{i}—Sn1—O2	90.4 (3)
C10—Sn1—O1^{i}	103.6 (3)	O1^{i}—Sn1—O2	132.4 (2)
C10—Sn1—O1	110.8 (3)	O1—Sn1—O2	54.6 (2)
O1^{i}—Sn1—O1	78.7 (3)	O2—Sn1—O2^{i}	173.0 (3)
C10—Sn1—O2	86.9 (3)		

Symmetry code: (i) $2 - x, 2 - y, z$.

All H atoms were positioned geometrically and refined as riding on their parent atoms, with aromatic C—H distances of 0.93 Å, methylene C—H distances of 0.97 Å and methyl C—H distances of 0.96 Å. The U_{iso}(H) values were set at $1.5U_{eq}$(C) for the methyl H atoms and at $1.2U_{eq}$(C) for the other C-bound H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

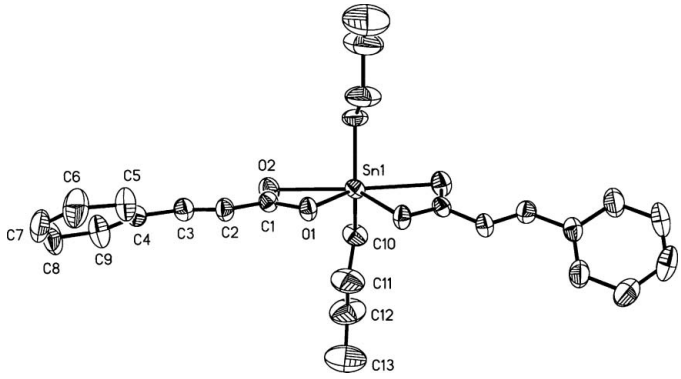


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. Unlabeled atoms are related to labeled atoms by the symmetry code $(2 - x, 2 - y, z)$.

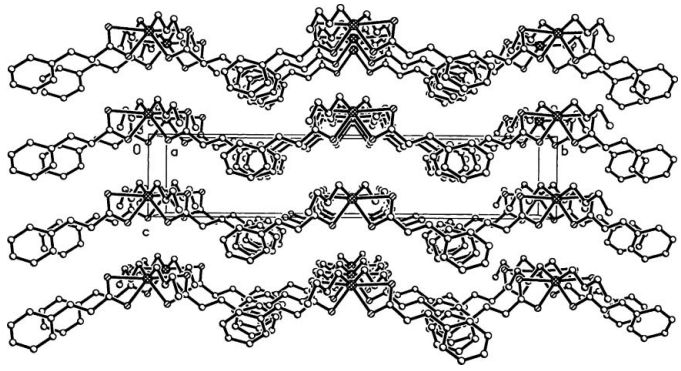


Figure 2
The crystal packing of the title complex. H atoms have been omitted.

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