

## References

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## {2-[(2,3-Dimethylphenyl)amino]benzoato-O:O'}trimethyltin(IV)

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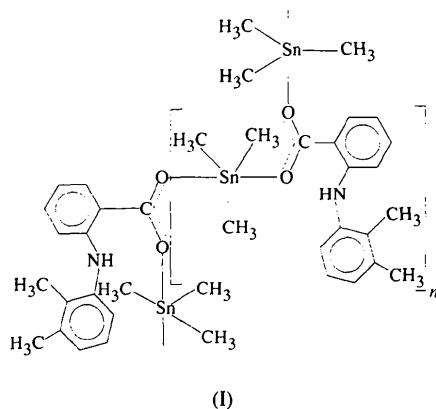
## Abstract

In the title compound,  $[\text{Sn}(\text{C}_{15}\text{H}_{14}\text{NO}_2)(\text{CH}_3)_3]$ , the Sn atom has a distorted trigonal bipyramidal coordination. The three bonds to methyl groups in the equatorial plane have almost the same values [Sn—C range 2.106(3)–2.113(4) Å], but the Sn—O bonds in the axial positions involving one carboxyl O atom [Sn1—O1 2.153(2) Å] and another symmetry-related carboxyl O atom in the *trans* position [Sn1—O2 2.495(2) Å] have quite different values. The O1—Sn1—O2 angle is 173.60(8)°. Each trimethyltin group bridges two neighbouring 2-[(2,3-dimethylphenyl)amino]benzoate ligands via carboxyl moieties to form polymeric chains.

## Comment

Organotin carboxylates containing a six-membered ring with a heteroatom either as part of the ring skele-

ton or as an additional functional group, have various structural possibilities. Such variations depend on the nature of the heteroatom. If the heteroatom is a potential donor ligand, like N, O or S, it increases the coordination number of the Sn atom, either intramolecularly or by forming an intermolecular interaction with the Sn atom of a symmetry-related molecule. The known examples with nitrogen as the heteroatom are: dimethylchlorotin 2-pyridinecarboxylate (Nowell, Brooks, Beech & Hill, 1983), dicarboxylatotetraorganodistannoxane  $\{[{}^n\text{Bu}_2\text{Sn}(\text{O}_2\text{CC}_5\text{H}_4\text{N})]_2\text{O}\}_2$  (Parulekar *et al.*, 1989) and three  $\text{Me}_2\text{Sn}(\text{chelate})_2$  compounds bearing five-membered chelate rings (Lockhart & Davison, 1987). In the last case, when the heteroatom is away from the  $\text{CO}_2$  group (either included or attached to the ring), the intermolecular interactions result in the formation of an infinite polymeric chain, at least in the solid state, *e.g.* trimethylstannyl 2-furancarboxylate (Tiekink, Sandhu & Verma, 1989). In the present case, however, the heteroatom (in the form of an amino group) connects two six-membered rings, namely the benzoate and xylyl groups, but is itself located on the C atom adjacent to the  $\text{CO}_2$  group. The structure of the title compound, (I), was determined in order to study the influence of the N atom on the coordination number of the Sn atom.



As can be seen from Fig. 1, the central five-coordinated Sn atom has a distorted trigonal bipyramidal environment. The three methyl groups are located in the basal plane and the more electronegative O atoms from symmetry-related carboxylate ligands occupy the axial positions. The Sn atom is 0.153(2) Å out of the equatorial plane towards the more strongly bound O1 atom. The three Sn—C distances are equal within experimental error [2.106(3), 2.113(4) and 2.109(3) Å] and are also in agreement with the values reported for related compounds. The Sn—O bond lengths are significantly different [Sn1—O1 2.153(2) and Sn1—O2 2.495(2) Å]. The C—O bonds within the carboxyl group also have different lengths. The longer C4—O1 bond [1.292(4) Å] and the shorter Sn—O1 bond [2.153(2) Å] share the same O atom and *vice versa*.

This could be interpreted so that one of the carboxyl O atoms forms a covalent bond with the Sn atom, but the other carboxyl O atom (double bond) is coordinated to tin with a longer Sn—O distance because the C—O bond keeps some of its double-bond nature. The O1—Sn1—O2 angle is 173.60(8)°. The central trimethyltin group bridges two neighbouring 2-[(2,3-dimethylphenyl)amino]benzoate ligands *via* carboxylate moieties to form one-dimensional polymeric chains (Fig. 2) running along the *a* axis. Similar polymeric chain structures of triorganotin compounds with carboxylate bridges

are also observed in trimethylstannyl 2-furancarboxylate (Tiekink, Sandhu & Verma, 1989), dimethylchlorotin 2-pyridinecarboxylate (Nowell, Brooks, Beech & Hill, 1983) and triorganotin 2-(2-thiophenyl)ethylene-1-carboxylate derivatives (Danish *et al.*, 1995). A two-dimensional polymeric structure where the carboxylate group bonds two organometallic moieties in a bridging bidentate fashion is reported for poly[*trans*-bis(trimethyltin(IV))- $\mu$ -(2,2'-bipyridyl-4,4'-dicarboxylato-*O*:*O'*:*O''*:*O'''*)] (Stocco *et al.*, 1996). The two six-membered rings in the 2-[(2,3-dimethylphenyl)amino]benzoate ligand are planar with a dihedral angle of 112.5(2)° between them. The dihedral angles between the carboxyl group and the two rings are 9.4(2)° for phenyl and 113.5(2)° for xylyl. Bond distances and angles within the ligands have their expected values. There exists an intramolecular N1—H1...O1 hydrogen bond with *D*—A 2.635(3) Å and *D*—H...A 137.08(2)°.

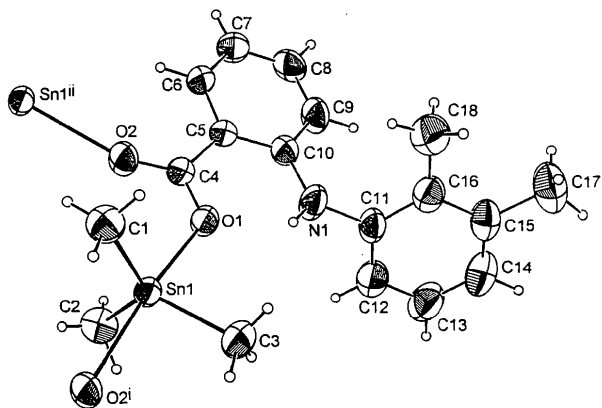


Fig. 1. ORTEP (Johnson, 1976) drawing of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

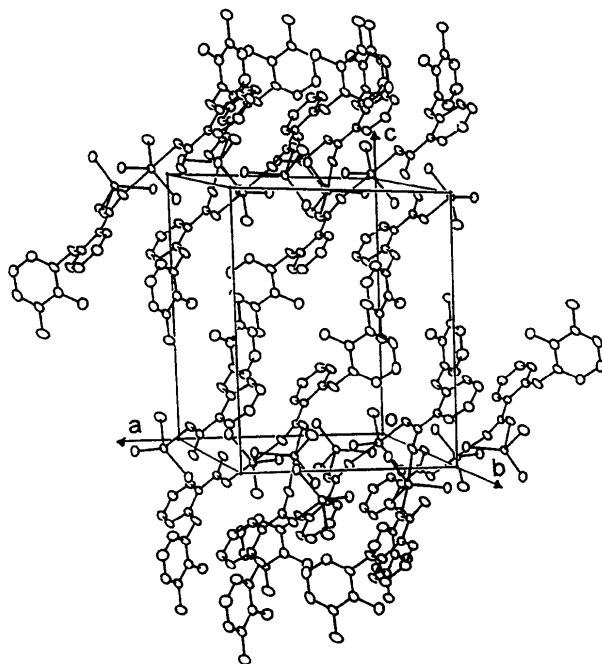


Fig. 2. Packing diagram showing the polymeric chains running along the *b* axis.

## Experimental

Since most of the organotin halides and their carboxylate derivatives are air and moisture sensitive, all the glassware was completely dried at 413 K. The reaction was carried out in dried solvents under argon. The title compound was prepared by the reaction of (CH<sub>3</sub>)<sub>3</sub>SnCl and the silver salt of 2-[(2,3-dimethylphenyl)amino]benzoic acid in a 1:1 molar ratio.

## Crystal data

[Sn(C<sub>15</sub>H<sub>14</sub>NO<sub>2</sub>)(CH<sub>3</sub>)<sub>3</sub>]

*M<sub>r</sub>* = 404.08

Monoclinic

*P*2<sub>1</sub>/*a*

*a* = 12.506(2) Å

*b* = 9.778(2) Å

*c* = 14.995(3) Å

β = 95.43(5)°

*V* = 1825.4(6) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.47 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10.33–18.16°

μ = 1.41 mm<sup>-1</sup>

*T* = 295 K

Prismatic

0.45 × 0.25 × 0.20 mm

Colourless

## Data collection

Enraf–Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction: empirical *via* ψ scans (*MoLEN*; Fair, 1990)

*T<sub>min</sub>* = 0.628, *T<sub>max</sub>* = 0.754

3007 measured reflections

2769 independent reflections

2253 reflections with *I* > 3σ(*I*)

*R<sub>int</sub>* = 0.012

θ<sub>max</sub> = 23.57°

*h* = -14 → 13

*k* = 0 → 10

*l* = 0 → 16

3 standard reflections

frequency: 120 min

intensity decay: 2.52%

## Refinement

Refinement on *F*

*R* = 0.021

*wR* = 0.029

(Δ/σ)<sub>max</sub> = 0.002

Δρ<sub>max</sub> = 0.489 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.248 e Å<sup>-3</sup>

$S = 1.16$   
2167 reflections  
199 parameters  
H atoms: see below  
Weighting scheme: see below

Extinction correction: none  
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Stocco, G., Gul, G., Giraslo, M. A., Bruno, G., Nicolo, F. & Scopelliti, R. (1996). *Acta Cryst. C* **52**, 829–832.  
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Table 1. Selected geometric parameters (Å, °)

|                         |            |                          |           |
|-------------------------|------------|--------------------------|-----------|
| Sn1—O1                  | 2.153 (2)  | O2—C4                    | 1.242 (4) |
| Sn1—O2 <sup>i</sup>     | 2.495 (2)  | N1—C10                   | 1.368 (4) |
| Sn1—C1                  | 2.106 (3)  | N1—C11                   | 1.437 (4) |
| Sn1—C2                  | 2.113 (4)  | C4—C5                    | 1.480 (4) |
| Sn1—C3                  | 2.109 (3)  | C5—C6                    | 1.394 (4) |
| O1—C4                   | 1.292 (4)  | C5—C10                   | 1.417 (4) |
| O1—Sn1—O2 <sup>i</sup>  | 173.60 (8) | C1—Sn1—C3                | 113.9 (2) |
| O1—Sn1—C1               | 97.5 (1)   | C2—Sn1—C3                | 119.3 (1) |
| O1—Sn1—C2               | 96.3 (1)   | Sn1—O1—C4                | 123.8 (2) |
| O1—Sn1—C3               | 88.1 (1)   | Sn1 <sup>ii</sup> —O2—C4 | 162.3 (2) |
| O2 <sup>i</sup> —Sn1—C1 | 88.3 (1)   | C10—N1—C11               | 125.9 (3) |
| O2 <sup>i</sup> —Sn1—C2 | 82.4 (1)   | O1—C4—C5                 | 117.1 (2) |
| O2 <sup>i</sup> —Sn1—C3 | 87.1 (1)   | O2—C4—C5                 | 121.8 (3) |
| C1—Sn1—C2               | 125.2 (1)  | O1—C4—O2                 | 121.1 (3) |

Symmetry codes: (i)  $\frac{3}{2} - x, y - \frac{1}{2}, 2 - z$ ; (ii)  $\frac{3}{2} - x, \frac{1}{2} + y, 2 - z$ .

The weighting scheme used was  $w = 4F^2/[\sigma(I)^2 + (0.04F^2)^2]$ , if  $F^2 < 4[\sigma(I)^2 + (0.04F^2)^2]^{1/2}$  then the reflection is omitted. All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their corresponding C atoms of the phenyl and xyllyl rings, while the H atoms of N1, C17 and C18 were taken from a difference map and refined for a few cycles. For all H atoms, a riding model was used with  $B_{eq}(H) = 1.3B_{eq}(C, N)$ .

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: NA1265). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tetracarbonyl( $\eta^{2,2}$ -1,5-cyclooctadiene)-molybdenum(0)

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## Abstract

In the title compound, tetracarbonyl[(1,2,5,6- $\eta$ )-1,5-cyclooctadiene]molybdenum(0),  $[\text{Mo}(\text{C}_8\text{H}_{12})(\text{CO})_4]$ , the coordination polyhedron around the metal atom is a distorted octahedron. The Mo—C bond distance for CO groups *trans* to a C=C unit is significantly shortened [average value 1.948 (8) Å], while the Mo—C bond distances for the *cis* groups are only slightly shortened [average value 2.031 (8) Å], compared with the values reported for  $[\text{Mo}(\text{CO})_6]$ . The average distance of the Mo atom to the midpoints of the olefinic C=C bonds is 2.388 (8) Å.

## Comment

Thermal or photochemical reactions between hexacarbonylmolybdenum(0) and 1,5-cyclooctadiene (COD) form the olefin complexes of the group 6 metals  $[\text{M}(\text{CO})_4(\eta^{2,2}\text{-COD})]$  (Fischer & Fröhlich, 1959; Dixon, Kola & Howell, 1984). Olefin complexes have been used as powerful  $[\text{M}(\text{CO})_4]$  transfer reagents in the syntheses of many transition metal carbonyl derivatives due to the lability of the metal–diene bond (King & Fronzaglia, 1966). The stereochemistry of the rigid complexes has been studied by IR (Darensbourg, Tappan & Nelson, 1977) and <sup>13</sup>C NMR spectroscopy (Darensbourg, Nelson & Murphy 1977; Kotzian, Kreiter & Özkaz, 1982; Gryf-Keller, Krawczyk & Szczecinski, 1991). Despite the extensive use of the  $[\text{Mo}(\text{CO})_4(\eta^{2,2}\text{-diene})]$  complexes, only the structure of the analogous tungsten compound with norbornadiene (Greveles, Jacke, Betz, Krüger & Tsay, 1989) appears to have been reported. The present paper deals with the structure determination of  $[\text{Mo}(\text{CO})_4(\eta^{2,2}\text{-COD})]$ , (I), and the interpretation of the IR and <sup>13</sup>C NMR spectroscopic data.