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cis-Bis(8-hydroxyquinolinato-κ^2N,O)diphenyltin(IV)

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The coordination geometry at the Sn atom in the title compound, $[Sn(C_6H_5)_2(C_9H_6NO)_2]$, is distorted *cis*-octahedral, in which the two phenyl ligands are *cis* to one another and *trans* to the N atoms of the oxinate ligands. This arrangement brings the oxinate O atoms into a *trans* relationship and is the geometry adopted most frequently by diorganotin(IV) oxinates. The O···O vector in the molecule is almost parallel to the plane of every ligand.

Key indicators

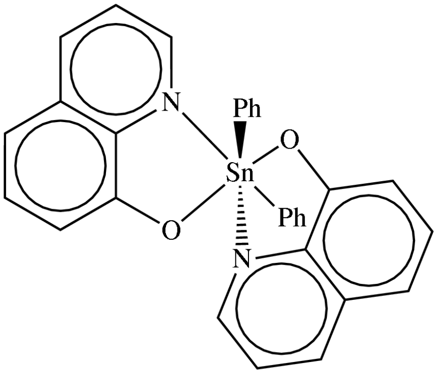
Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.091
Data-to-parameter ratio = 22.6

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

Comment

Diorganotin(IV) complexes have been studied extensively because of their interesting chemical, biological and pharmaceutical properties. The relationship between the structure and anti-tumour activity of di- and triorganotin(IV) oxinates and thiooxinates has been discussed by Gielen *et al.* (1993). A brief overview of the molecular structures and the coordination geometry at the Sn atom adopted by diorganotin(IV) bischelates is given by Ng *et al.* (1987) and Kellö *et al.* (1995). The latter paper points out the difficulty of determining the relative stereochemical arrangement of the ligands at the Sn atom from spectroscopic measurements alone, and the consequent need for crystal structure determinations. The solid-state molecular structures of several diorganotin(IV) dioxinates, $[R_2Sn(oxin)_2]$, where oxinH = 8-hydroxyquinoline, have been studied by Mössbauer spectroscopy (Kumar Das *et al.*, 1981), NMR spectroscopy (Lockhart & Manders, 1987) and X-ray diffraction.

The Cambridge Structural Database (July 2004 version; Allen, 2002) contains data for seven complexes of the type $[R_2Sn(oxin)_2]$, where R_2 is dimethyl (Schlemper, 1967), bis(*O*-ethylxanthato) (Raston *et al.*, 1978), dichloro (Archer *et al.*, 1987), di-*n*-butyl (Shi & Hu, 1988), *p*-chlorophenyl-*p*-tolyl (Chen *et al.*, 1989), *n*-butyl-chloro (Kellö *et al.*, 1995) and bis(2-methoxycarbonylethyl) (Ng & Kumar Das, 1995). The structure of the complex where R_2 is methyl-bis(trimethylsilyl)methyl is also known (Ng, 1981). The structure of an ethyl-*n*-propyl derivative has also been reported, where the oxinate ligands are the 2-methyl-substituted 8-hydroxyquinolinate (Kumar Das *et al.*, 1984). All of the compounds crystallize as molecular chelate complexes, where the oxinate ligands have bidentate functionality. In all but one case, the spatial arrangement of the ligands around the central Sn atom is distorted *cis*-octahedral, in which the two R ligands are *cis* to one another and *trans* to the N atoms of the oxinate ligands. This arrangement brings the oxinate O atoms into a *trans* relationship. The exception is the ethyl-*n*-propyl derivative with 2-methyl-substituted 8-hydroxyquinolinate ligands (Kumar Das *et al.*, 1984), in which the R ligands adopt positions that are closer to being *trans* to one another and the four chelating oxinate atoms are coplanar with the Sn atom, with the O atoms in *cis* positions. This arrangement generates a skew-trapezoid bipyramidal coordination geometry. The departure from the usual *cis*-octahedral coordination geometry was attributed to the steric effects of the 2-methyl substituent on the oxinate ligands.



(I)

The crystal structure of the title complex, (I), has been determined in order to investigate the effect on the coordination geometry at the Sn atom of the more bulky phenyl group as the *R* ligands. A view of the molecular structure of (I) is shown in Fig. 1. Selected geometric parameters, which describe the coordination geometry about the Sn atom, are given in Table 1. Complex (I) adopts the usual distorted *cis*-octahedral coordination geometry, with exactly the same arrangement of donor atoms as found in the previous structures of this type. The Sn—C, Sn—N and Sn—O bond lengths, the bond angles at the Sn atom and the bite angles of the oxinate ligands are not significantly different from the previously observed values in those compounds where both *R* ligands coordinate to the Sn atom *via* C atoms. Therefore, the phenyl ligands do not influence the coordination geometry any differently than the *R* ligands in the other reported $[R_2Sn(oxin)_2]$ structures.

The oxinate ligands in (I) are essentially planar and these planes have a dihedral angle of 83.22 (7)°. The O1· · ·O2 vector in the molecule is almost parallel to the plane of every ligand, with the phenyl ring defined by atoms C19–C24 being the most tilted. The angles between the O1· · ·O2 vector and the planes of the oxinate ligands containing atoms N1 and N2 are 4.00 (7) and 4.01 (7)°, respectively. The angles between the O1· · ·O2 vector and the planes of the phenyl rings defined by atoms C19–C24 and C25–C30 are 17.57 (10) and 6.89 (11)°, respectively.

Experimental

A hot anhydrous benzene solution of 8-hydroxyquinoline (0.18 g, 1.29 mmol) was added dropwise to a stirred benzene solution containing Ph_3SnCl (0.5 g, 1.29 mmol) and refluxed for 2 h. Et_3N (0.130 g, 1.29 mmol) was added to the reaction mixture, which was refluxed for an additional 1.5 h and cooled to room temperature. After removing $Et_3N·HCl$ by filtration, the filtrate was allowed to evaporate at room temperature, which afforded pale-yellow crystals. Subsequent crystallization from benzene yielded the title compound, (I) (55% yield, m.p. 505–506 K). Analysis calculated for $C_{30}H_{22}N_2O_2Sn$: C 64.20, H 3.95, N 4.99%; found: C 64.10, H 3.90, N 5.01%. The analytical data and m.p. of (I) correspond well with those of the product obtained by another synthetic route (Blake *et al.*, 1961). X-ray diffraction quality crystals were grown by slow evaporation of a solution of (I) in acetone.

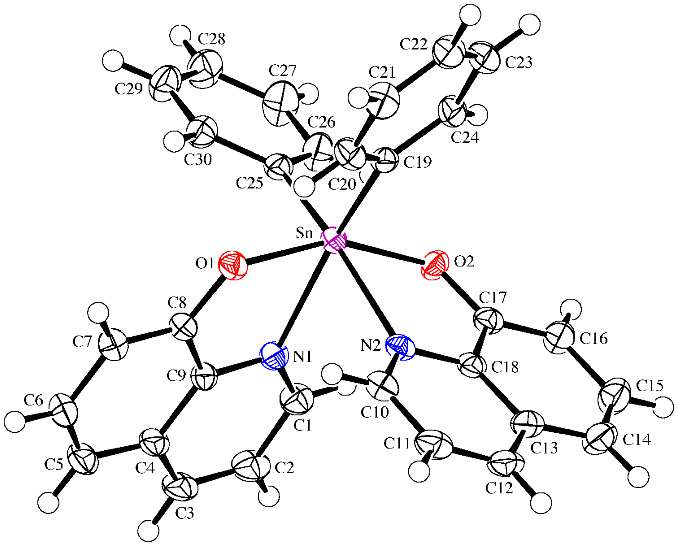


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary size.

Crystal data

$[Sn(C_6H_5)_2(C_9H_6NO)_2]$
$M_r = 561.12$
Monoclinic, $P2_1/n$
$a = 12.8111 (3)$ Å
$b = 14.8721 (4)$ Å
$c = 12.8378 (3)$ Å
$\beta = 91.3964 (14)°$
$V = 2445.23 (10)$ Å^3
$Z = 4$

$D_x = 1.524$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 90 230 reflections
$\theta = 2.0–30.0°$
$\mu = 1.07$ mm^{-1}
$T = 160 (1)$ K
Tablet, yellow
0.25 × 0.25 × 0.07 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans with κ offsets
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.697$, $T_{max} = 0.933$
68 827 measured reflections

7135 independent reflections
5326 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 30.0°$
$h = -16 \rightarrow 18$
$k = -20 \rightarrow 20$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.091$
$S = 1.06$
7130 reflections
316 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 2.0301P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 1.67$ e Å^{-3}
$\Delta\rho_{min} = -1.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn—O1	2.0809 (18)	Sn—N2	2.327 (2)
Sn—O2	2.0812 (18)	Sn—N1	2.331 (2)
Sn—C25	2.154 (3)	O1—C8	1.336 (3)
Sn—C19	2.155 (2)	O2—C17	1.328 (3)
O1—Sn—O2	156.94 (7)	C25—Sn—N2	161.11 (9)
O1—Sn—C25	100.92 (9)	C19—Sn—N2	87.78 (8)
O2—Sn—C25	92.04 (9)	O1—Sn—N1	74.59 (7)
O1—Sn—C19	91.72 (8)	O2—Sn—N1	86.68 (7)
O2—Sn—C19	102.19 (8)	C25—Sn—N1	89.42 (8)
C25—Sn—C19	108.61 (9)	C19—Sn—N1	159.36 (9)
O1—Sn—N2	87.53 (7)	C1—N1—Sn	129.73 (18)
O2—Sn—N2	74.90 (7)	C10—N2—Sn	130.15 (19)

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with a C—H distance of 0.95 Å and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Five low-angle reflections were omitted from the final cycles of refinement because their observed intensities were much lower than the calculated values, as a result of being partially obscured by the beam stop. The two largest peaks of residual electron density are 1.67 and 1.61 e Å^{-3} (next peak is 0.69 e Å^{-3}) and lie less than 0.9 Å from the Sn and O1 atoms, respectively. The deepest hole in the residual electron density map is 0.8 Å from the Sn atom.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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