CRYSTAL STRUCTURE OF DIPHENYLTIN(IV) BIS(TETRAPHENYLIMIDODIPHOSPHINATE)

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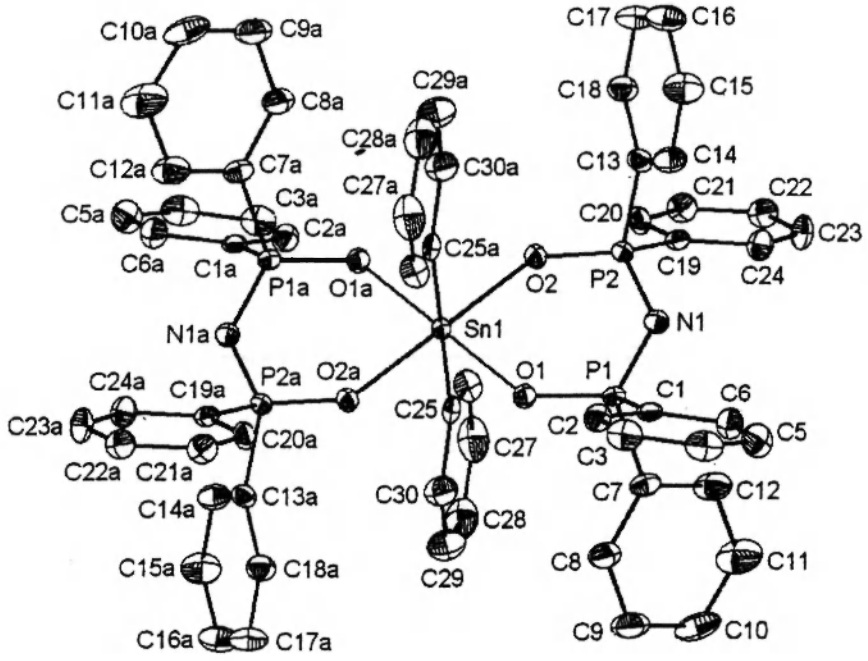

Figure 1. Molecular structure of **I** (hydrogen atoms are omitted for clarity): Selected bond distances (Å) and angles (°): Sn1–O1 2.182(2), Sn1–O2 2.170(2), Sn1–C25 2.124(4), O1–P1 1.516(3), O2–P2 1.525(3), P1–N1 1.586(3), P2–N1 1.580(3), O1–Sn1–O2 90.88(9), O1–Sn1–O2a 89.12(9), C25–Sn1–C25a 180.0(3). Symmetry operation given by "a": $-x + 2, -y, -z$.

COMMENT

Although several Main Group metal derivatives of the tetraphenylimidodiphosphinato ligand, $[(OPPh_2)_2N]^-$, are known,[1] just a few compounds containing organotin(IV) and organolead(IV) fragments were investigated by X-ray diffraction.[2-4] The mononuclear organotin(IV) complex $Ph_2Sn[(OPPh_2)_2N]_2$ (**I**) crystallizes in monoclinic space group $P2_1/n$ and has an inversion centre (Fig. 1). The imidodiphosphinato ligands are isobidentate [Sn1–O1 2.182(2), Sn1–O2 2.170(2), O1–P1 1.516(3) and O2–P2 1.525(3) Å]. In the spiro-bicyclic $NP_2O_2SnO_2P_2N$ system the *trans* O–Sn–O angles are 180.0(2)°, while *cis* O–Sn–O angles are 90.88(9)° (endocyclic) and 89.12(9)° (exocyclic), resulting in a co-planar SnO_4 system [Σ(O–Sn–O) = 360°]. The two Sn–C bonds are co-linear [C25–Sn1–C25a 180.0(3)°] and perpendicular to the SnO_4 plane (C–Sn–O range 89.4 - 90.6°). The resulting coordination polyhedron of the central tin atom is an almost perfect *trans*-octahedron, similar to those reported for $Bu_2Sn[(OPPh_2)_2N]_2$[2] and $Me_2Sn[(SPPh_2)_2N]_2$[5], but less distorted.

EXPERIMENTAL[1]

The compound was prepared as in ref. 2. Crystals suitable for single-crystal X-ray diffraction were obtained from a CH_2Cl_2 / n-hexane mixture (1:4 volume ratio). The crystal structure measurement and refinement data for I are given in Table 1. Data for I were collected using a SMART APEX diffractometer ("Babes-Bolyai" University) at 297 K. A graphite monochromator was used to produce a wavelength (Mo-$K\alpha$) of 0.71073 Å. The structure was solved by direct methods (full-matrix least-squares on F^2). All non-hydrogen atoms were refined with anisotropic thermal parameters.

Tabel 1. Crystal data and structure refinement for $Ph_2Sn[(OPPh_2)_2N]_2$

Empirical formula	$C_{60}H_{50}N_2O_4P_4Sn$	Formula mass	1105.60
Crystal system	monoclinic	Space group	P2(1)/n
a [Å]	15.2086(16)	b [Å]	9.6128(10)
c [Å]	18.3934(19)	α [°]	90
β [°]	103.615(2)	γ [°]	90
Z	2	V [Å^3]	2613.5(5)
F(000)	1132	Crystal size [mm]	0.52 x 0.29 x 0.08
$D_{calcd.}$ [g cm^{-3}]	1.405	θ range [°]	1.57 to 26.02
No. of measured data	19924	No. of unique data [R(int)]	5145 [R(int) = 0.0594
Data/restraints/parameters	5145 / 0 / 322	Absorption correction	multi-scan
R_1 [$I > 2\sigma(I)$]	0.0541	wR_2 [$I > 2\sigma(I)$]	0.1060
R_1 (all data)	0.0682	wR_2 (all data)	0.1120
Goodness-of-fit on F^2	1.144	Residual density [e·Å^{-3}]	1.097 and -0.998
Diffractometer	Bruker AXS SMART APEX CCD	Programs used	SMART,[6] SAINT,[6] SHELXTL[6] and Diamond 3[7]
Deposition number	CCDC-277624		

Acknowledgments

This work was supported by the National University Research Council of Romania (CNCSIS grant, A$_T$-6/33/2005, and CERES Project, Contract No 32/12.11.2004). We thank the *National Center for X-Ray Diffraction* ("Babes-Bolyai" University, Cluj-Napoca) for support of the single-crystal X-ray diffraction studies.

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