CRYSTAL STRUCTURE OF [2,6-BIS(DIMETHYLAMINOMETHYL)PHENYL]DIPHENYLTIN HEXAFLUOROPHOSPHATE: $\{(C_6H_5)_2Sn[C_6H_3(CH_2NMe_2)_2\text{-}2,6\}^+ PF_6^-$

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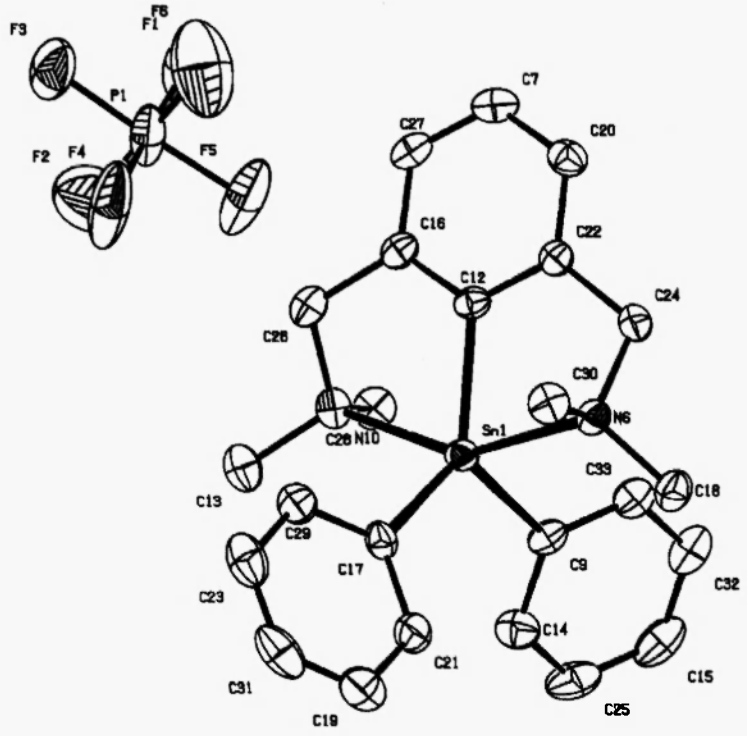

Figure 1. The molecular structure of $\{(C_6H_5)_2Sn[C_6H_3(CH_2NMe_2)_2\text{-}2,6\}^+ PF_6^-$ (50% probability ellipsoids). Selected bond lengths (Å) and angles (°): Sn1-C1 2.092(3), Sn1-C21 2.122(3), Sn1-C31 2.131(3), Sn1-N1 2.415 (2), Sn1-N2 2.410 (2), F-P (average) 1.585, C1-Sn1-C21 126.6 (1), C21-Sn1-C31 115.0 (1), C1-Sn1-C31 118.26 (1), N1-Sn1-N2 152.03 (8).

Comment

The structure [2,6-bis(dimethylaminomethyl)phenyl]diphenyltin hexafluorophosphate consists of PF_6^- ion interspersed among the $\{(C_6H_5)_2Sn[C_6H_3(CH_2NMe_2)_2\text{-}2,6\}^+$ cation in the unit cell. The hexafluorophosphate anion is out of the primary co-ordination sphere of central tin atom. The ligand is bonded and twice co-ordinated, respectively, in the tridentate fashion to the central tin(IV) atom through the C and two N atoms. The pseudo trigonal bipyramidal geometry about the tin atom is defined by the three donor atoms from the terdentete ligand and two ipso carbon atomsderived from the phenyl groups so that the three carbon atoms define the trigonal plane. The triorganotin cation has a planar (ΣC-Sn-C =359.9(3)°) C_3Sn girdle and the distortion of the *trans*-trigonal bipyramidal geometry results from the bent N-Sn-N (angle 152.03(8)°) skeleton. These general features of the cation are close to its bromide salts studied previously[1]. However, from the more detailed comparison follows, that some parameters of tin coordination sphere differ significantly, especially Sn-N distances and angles in C_3Sn girdle being : Sn1-N1 2.440(1)Å, C11-Sn1-C21 124.95(4)°, C1-Sn1-C31 124.95(4)°, C21-Sn1-C31 110.09(9)° for Br^- salt (Symmetry code:(i) -x, y, 3/2-z).

Experimental

All preparations were carried out under anaerobic conditions. The final compound was prepared from starting [2,6-bis(dimethylaminomethyl)phenyl]diphenyltin bromide and KPF_6 in dichloromethane. The single crystals were carried out upon vapour diffusion of n-hexane into dichloromethane solution of

above mentioned compound. Yield: 0.31g (55%); m.p. 195-205°C. (Found: C, 47.35; H, 4.83; N, 4.56. $C_{24}H_{29}N_2SnPF_6$ requires C, 47.32; H, 4.8; N, 4.6.). ^1H NMR ($CDCl_3$): δ 7.63 (d, aryl-H, 4H), 7.59 (m, aryl-H, 9H), 3.97 (s, NCH_2-H, 4H), 2.21 (s, CH_3-H, 12H). Negative-ion ESI-MS: m/z 145, exp. 100% (theor. 100%). The cationic part of the molecule ($C_{24}H_{29}N_2Sn$) with characteristic tin isotopes was measured in the positive ion mode (theoretical relative abundances in parenthesis): m/z 461, 23.9% (40.8%); m/z 462, 21.7% (32.7%); m/z 463, 59.5% (74.9%); m/z 464, 34.4% (43.6%); m/z 465, 100% (100%); m/z 466, 24.5% (26.4%); m/z 467, 13.3% (16.4%); m/z 468, 3.7% (3.8%); m/z 469, 17.0% (16.6%) and m/z 470, 4.3% (4.5%)..

X-Ray Crystallography

The colourless crystal of suitable dimensions was mounted on glass fibbers with epoxy cement and measured on four-circle diffractometer Nonius KappaCCD with CCD area detector at 150(2)K with MoKα radiation. The crystallographic details are summarised in Table 1. The structures were solved by the direct method (SIR97)[2] and refined by a full matrix least squares procedure based on F^2 (SHELXL97)[3-5]. The absorption was corrected by Gaussian method during data reduction. All hydrogen atoms were localised on a difference Fourier map and refined isotropically. The final difference map had no peaks of chemical significance. Scattering factors were those implemented in the SHELX programs.

Table 1. Crystal data for $\{(C_6H_5)_2Sn[C_6H_3(CH_2NMe_2)_2\text{-}2,6\}^+ PF_6^-$

Formula	$C_{24}H_{29}F_6N_2PSn$	Formula weight	609.15
Crystal system	Monoclinic	Crystal size, mm	0.32x0.09x0.08
Space group	$P\,2_1/c$	a, Å	11.1090(2)
b, Å	19.8550(3)	c, Å	11.8400(2)
β, °	105.015(1)	V, Å^3	2522.38(7)
Z	4	Diffractometer	Nonius KappaCCD
Temperature, K	150(2)	μ(Mo-Kα), cm^{-1}	11.36
Trans. factors	0.928-0.744	D_{calcd}, g cm^{-3}	1.604
F(000)	1224	θ_{max}	27.87
Reflns meas.	52291	Reflns unique, R_{int}	6013, 0.071
Reflns with $I \geq 2.0\sigma(I)$	4269	w_1;w_2[b]	0.0429;1.0999
$R(F)$[a], $R_w(F^2)$[a]	0.035, 0.087	Δρ, e Å^{-3}	0.800; -0.778
GOF[a]	1.029	Deposition number	CCDC 164283
Program used	SIR97, SHELXL97, PLATON		

[a] Definitions : $R(F) = \Sigma||F_o|-||F_c||/\Sigma|F_o|$ for $I \geq 2.0\sigma(I)$, $wR2 = [\Sigma(w(F_o^2 - F_c^2)^2)/\Sigma(w(F_o^2)^2)]^{1/2}$, for all reflections, $GOF = [\Sigma(w(F_o^2 - F_c^2)^2)/(N_{refins} - N_{params})]^{1/2}$, [b] weighting scheme $w = [\Sigma^2(F_o^2) + (w_1P) + w_2P]^{-1}$; $P = [\max(F_o^2, 0) + 2F_c^2]/3$.

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