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Crystallographic report

Tribenzyltin(IV) azide

Anthony C. Burke, Mary F. Mahon and Kieran C. Molloy*

Department of Chemistry, University of Bath, Bath BA2 7AY, UK

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Tribenzyltin azide adopts a polymeric structure with 1,3-bridging azide groups; the tin exhibits an almost regular trigonal bipyramidal *trans*-N_2SnC_3 environment.

KEYWORDS: crystal structure; tin; azide

COMMENT

We have made extensive use of organotin azides in 3 + 2 cycloaddition reactions leading to organotin tetrazoles,[1] which generate remarkable supramolecular arrays.[2–4] As part of this study we have prepared tribenzyltin azide, which adopts a polymeric structure in which azide groups bridge tin centres in a 1,3 manner, i.e. using nitrogen centres at both ends of the azide group. With smaller hydrocarbon groups on tin, e.g. Me_3SnN_3,[5] 1,1-N_3 bridges are observed, whereas bulkier groups, e.g. $^tBu_3SnN_3$,[6] generate monomers. The structure of $(PhCH_2)_3SnN_3$ (Fig. 1) is intermediate between these extremes, which is reflected in the Sn–N bond lengths. In Me_3SnN_3, Sn–N and bridging Sn–N′ are equal (2.39 Å),[5] whereas in monomeric $^tBu_3SnN_3$ the absence of a bridging interaction serves to strengthen the only Sn–N bond (2.10 Å).[6] In contrast, 1,3-bridging in $(PhCH_2)_3SnN_3$ incorporates intermediate primary Sn–N(1) [2.16(1) Å] and Sn–N(3′) bridging

*Correspondence to: Kieran C. Molloy, Department of Chemistry, University of Bath, Bath BA2 7AY, UK.
E-mail: chskcm@bath.ac.uk

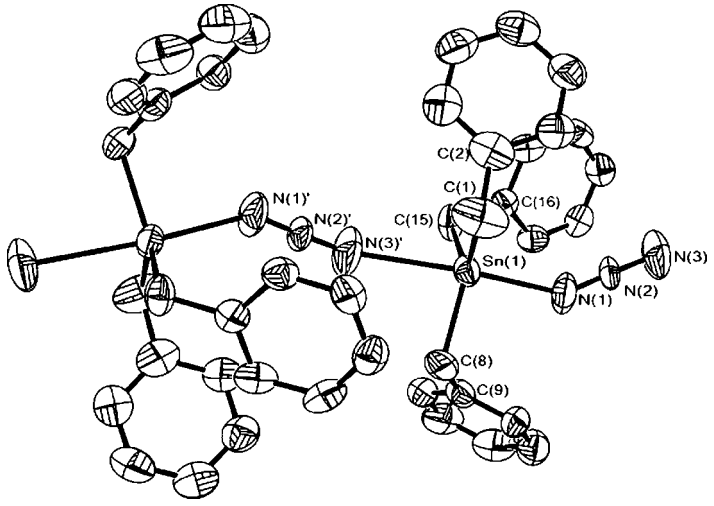

Figure 1. The structure of $(PhCH_2)_3SnN_3$; thermal ellipsoids are at the 30% probability level. Selected geometric data: Sn(1)–N(1) 2.16(1), Sn(1)–N(3′) 2.55(1), N(1)–N(2) 1.08(1), N(2)–N(3) 1.09(1) Å; N(1)–Sn(1)–N(3′) 173.6(7), C(1)–Sn(1)–C(8) 120.5(5), C(1)–Sn(1)–C(15) 117.0(5), C(8)–Sn(1)–C(15) 119.5(5), C(1)–Sn(1)–N(1) 98.1(7), C(8)–Sn(1)–N(1) 90.9(5), C(15)–Sn(1)–N(1) 98.3(4), C(1)–Sn(1)–N(3′) 88.2(7), C(8)–Sn(1)–N(3′) 85.1(5), C(15)–Sn(1)–N(3′) 79.6(5), N(1)–N(2)–N(3) 178.0(2)°. Symmetry operation: $-x$, $y + 1/2$, $-z-3/2$.

[2.55(1) Å] interactions. Despite the difference in axial bond lengths, the coordination sphere about tin approaches a regular trigonal bipyramid [<N–Sn–N′: 173.6(7)°]. Interestingly, the only other triorganotin azide that generates a polymer by 1,3-N_3 bridges, Ph_3SnN_3, incorporates much longer Sn–N′ links (2.86 Å).[7] Presumably, whereas the bulk of the benzyl groups inhibits 1,1-N_3 interactions, the flexibility afforded by the inclusion of the CH_2 groups into the ligand, which are absent in the case of Ph_3SnN_3, still allows a comparatively close approach of monomers.

EXPERIMENTAL

$(PhCH_2)_3SnN_3$ was prepared following literature precedent from $(PhCH_2)_3SnCl$[8] and NaN_3 (yield 50%, m.p. 116–119 °C).[9] Anal. Found: C, 56.4; H, 4.77; N, 8.28. Calc. for $C_{21}H_{21}N_3Sn$: C, 58.1; H, 4.84; N, 9.69%. ^{119}Sn NMR ($CDCl_3$): 7.9 ppm; $\nu(N_3)$ 2079 cm^{-1}. Intensity data were collected at 293(2) K on a CAD4 automatic four-circle diffractometer using a colourless block $0.17 \times 0.20 \times 0.20$ mm. $C_{21}H_{21}N_3Sn$, $M = 434.10$, orthorhombic, $P2_12_12_1$, $a = 7.953(3)$, $b = 13.076(6)$, $c = 18.633(12)$ Å, $U = 1937.7(17)$ Å^3, $Z = 4$, 1981 independent data ($R_{int} = 0.063$), (θ_{max} 25.1°), $R = 0.064$ (all data), $wR = 0.129$ (all data), $\rho_{max} = 1.22$ e$^-$ Å^{-3} (near tin), Flack parameter 0.06(10). Software used: SHELX86, SHELX93, ORTEX. CCDC deposition number 204 891.

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