X-Ray Crystal Structure of Bis(2-methylquinolin-8-olato)(ethyl)n-propyltin(IV)

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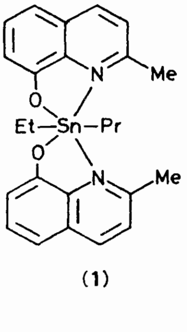
The crystal structure of the title compound, [Sn(Et)(Pr)($C_{10}H_8NO)_2$] (1), shows that the tin atom is octahedrally co-ordinated with the two alkyl groups approximately *trans* to each other (C–Sn–C 145°), in marked contrast to the well-documented *cis*-R_2Sn structure of bis(quinolin-8-olato)dimethyltin.

We have previously reported the observation based on Mössbauer spectral studies that the *cis*-R_2Sn skeletal geometry in octahedral dimethyl- and diphenyl-tin bis(quinolin-8-olate)s changes to a distorted *trans* configuration upon introducing a methyl substituent at the 2-position of the chelating ligand.[1] Supporting evidence for this has now been obtained from a crystallographic study of the mixed alkyl title complex, bis(2-methylquinolin-8-olato)(ethyl)n-propyltin (**1**).

Crystal data for (**1**): $C_{25}H_{28}N_2O_2Sn$, $M = 507.2$, monoclinic, space group $P2_1/n$, $a = 8.546(5)$, $b = 21.083(9)$, $c = 12.704(7)$ Å, $\beta = 94.39(3)°$, $U = 2282.23$ Å^3, $D_c = 1.48$ g cm^{-3} for $Z = 4$. 3362 Reflections were collected at room temperature on an Enraf-Nonius four-circle CAD-4 diffractometer with monochromatic Mo-K_α radiation. The structure was solved by heavy-atom methods and refined to a final R value of 5.3% for 2660 independent reflections with $F_o^2 > 3\sigma(F_o^2)$.†



(1)

Relevant structural data for the complex (see Figure 1) are listed in Table 1. The two alkyl groups are approximately *trans*

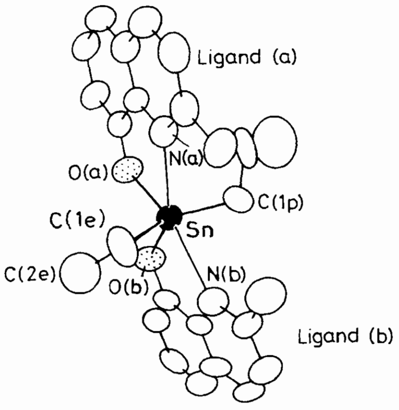


Figure 1

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† The atomic co-ordinates for this work are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Rd., Cambridge CB2 1EW. Any request should be accompanied by the full literature citation for this communication.

Table 1. Bond lengths (Å) and bond angles (°) with standard deviations in parentheses for bis(2-methylquinolin-8-olato)(ethyl)n-propyltin.[a]

Bond lengths			
Sn–C(1e)	2.145(8)	Sn–O(b)	2.066(4)
Sn–C(1p)	2.089(8)	Sn–N(a)	2.542(5)
Sn–O(a)	2.066(4)	Sn–N(b)	2.597(6)
Bond angles			
C(1e)–Sn–C(1p)	145.2(3)	C(1e)–Sn–O(a)	105.2(3)
N(a)–Sn–N(b)	139.3(2)	C(1e)–Sn–O(b)	101.6(3)
O(a)–Sn–O(b)	79.6(2)	C(1p)–Sn–N(a)	89.6(3)
O(a)–Sn–N(a)	70.9(2)	C(1p)–Sn–N(b)	78.2(3)
O(a)–Sn–N(b)	149.7(2)	C(1p)–Sn–O(a)	105.1(2)
C(1e)–Sn–N(a)	84.4(3)	C(1p)–Sn–O(b)	100.3(3)
C(1e)–Sn–N(b)	84.3(3)		

[a] The designations (a) and (b) serve to distinguish the chelating ligands, while C(1e) and C(1p) represent the carbon atoms of the ethyl and n-propyl groups, respectively, directly bonded to tin.

to each other [C(1e)–Sn–C(1p) 145.2(3)°] and perpendicular to the 2-methylquinolinolate groups. The latter are essentially coplanar with the donor oxygen atoms arranged *cis* to each other [O(a)–Sn–O(b) 79.6(2)°]. The shortest metal–ligand bonds are the Sn–O bonds [2.066(4) Å] while those to the ring nitrogens are the longest at 2.542(5) and 2.597(6) Å. This contrasts with the *cis*-arrangements of the methyl groups (110.7°) and of the donor nitrogen atoms (N–Sn–N 75.8°) in bis(quinolin-8-olato)dimethyltin,[2] with values of 2.11 ± 0.01 and 2.35 ± 0.04 Å respectively for the Sn–O and Sn–N bond distances. Also, a comparison of ligand 'bite angles' in these two complexes indicates that the values observed for the title complex are lower by *ca.* 3°. This situation would particularly favour a *cis*-structure on ligand–ligand repulsion model[3,4] considerations. It is therefore rather curious that this structure is not observed for the title complex. It seems to us highly unlikely that the *trans* preference of the EtSnPr moiety is the result of an over-play of electronic effects (as expressed by Bent's rule[5]) in the mixed alkyl complex relative to the dimethyltin case. A more plausible reason is that the unequal Sn–donor distances caused by steric factors in the quinolinolate ligand are better accomodated in an energetically more favourable distorted *trans*-structure than in a regular *cis*-structure.

The distorted *trans*-structure is also corroborated by the larger ^{119}Sn Mössbauer quadrupole splitting (Q.S.) value observed for the title complex (Q.S. = 3.17 ± 0.05 mm s^{-1}) relative to bis(quinolin-8-olato)(ethyl)n-propyltin (Q.S. = 2.09 mm s^{-1}). In general, Q.S. values for octahedral diorganotin(IV) derivatives increase with C–Sn–C angle opening and based on the point-charge model[6] the estimated value of this angle for the title complex is 133° (*vs.* 145° actually observed). Interestingly, for the bis(2-methylquinolin-8-olate) complexes of dimethylin and diphenyltin, the Q.S. values are larger at 3.27 and 3.50 mm s^{-1} respectively.[1]

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