# X-RAY STRUCTURE OF TRIS(DIISOBUTYLDITHIOCARBAMATO)PHENYLTIN(IV)

David J. Clarke,[1] Dainis Dakternieks,[2] and Edward R.T. Tiekink*[1]

[1]Department of Chemistry, The University of Adelaide, Australia 5005, edward.tiekink@adelaide.edu.au
[2]Centre for Chiral and Molecular Technologies, Deakin University, Geelong, Victoria 3217, Australia

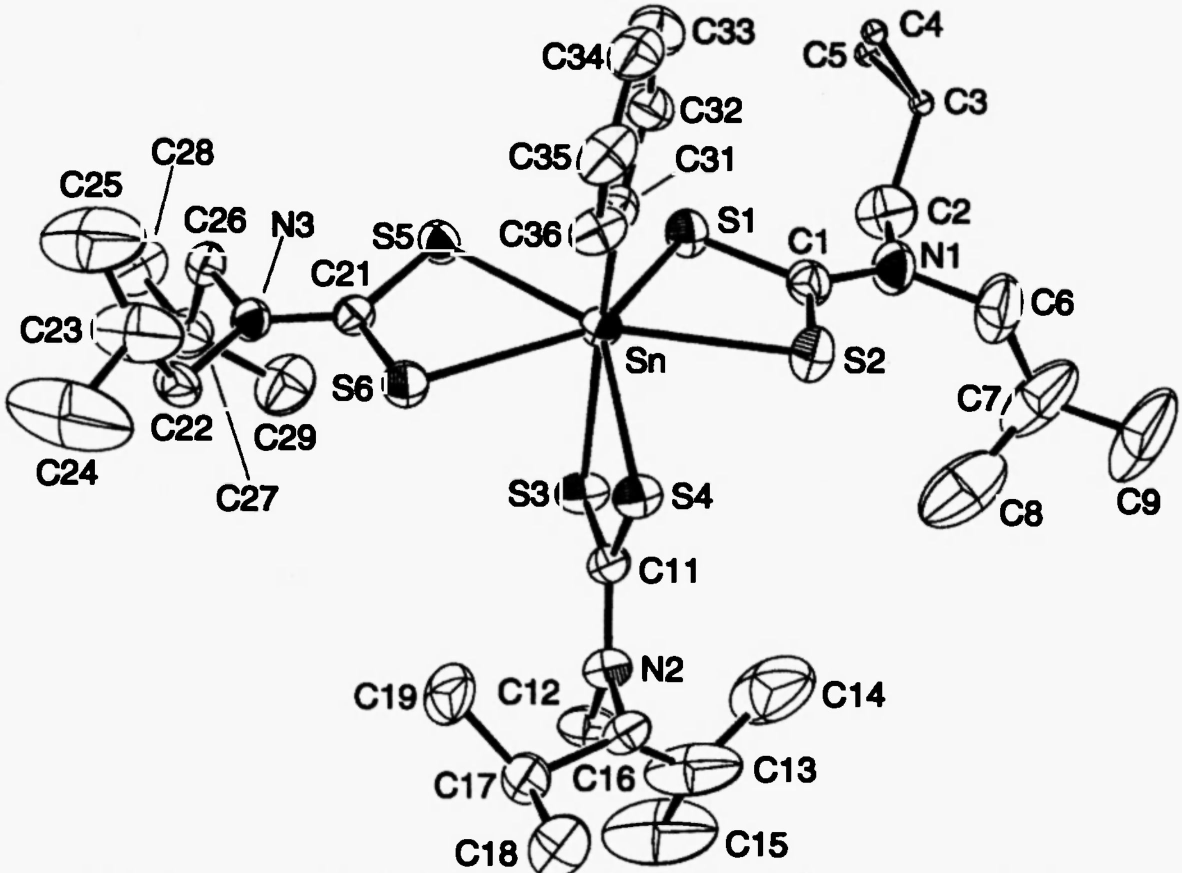

**Figure 1.** Molecular structure (50% displacement ellipsoids) of $[PhSn(S_2CN^iBu_2)_3]$. Selected bond distances and angles: Sn-S1 2.615(2), Sn-S2 2.709(1), Sn-S3 2.491(1), Sn-S4 2.729(1), Sn-S5 2.642(1), Sn-S6 2.774(2), Sn-C31 2.152(4), S1-C1 1.731(4), S2-C1 1.701(5), S3-C11 1.737(4), S4-C11 1.713(4), S5-C21 1.721(4), S6-C21 1.703(4), C1-N1 1.331(6), N2-C11 1.338(5), N3-C21 1.342(5) Å; S1-Sn-S2 66.49(4), S1-Sn-S3 91.13(4), S1-Sn-S4 138.20(4), S1-Sn-S5 76.32(3), S1-Sn-S6 141.07(4), S1-Sn-C31 101.0(1), S2-Sn-S3 87.34(4), S2-Sn-S4 75.92(4), S2-Sn-S5 142.75(4), S2-Sn-S6 150.66(4), S2-Sn-C31 91.5(1), S3-Sn-S4 68.90(4), S3-Sn-S5 91.60(4), S3-Sn-S6 83.09(4), S3-Sn-C31 166.2(1), S4-Sn-S5 137.57(4), S4-Sn-S6 74.76(4), S4-Sn-C31 97.5(1), S5-Sn-S6 65.45(4), S5-Sn-C31 97.6(1), S6-Sn-C31 91.4(1), Sn-S1-C1 89.7(2), Sn-S2-C1 87.2(2), Sn-S3-C11 90.0(1), Sn-S4-C11 82.9(1), Sn-S5-C21 90.1(1), Sn-S6-C21 86.1(2), S1-C1-S2 116.6(3), S3-C11-S4 118.2(2) and S5-C21-S6 117.7(2)°.

## Comment

The tin atom is coordinated by three bidentate dithiocarbamato ligands and the *ipso*-carbon atom of a phenyl group, and thereby exists in a $CS_6$ donor set that defines a distorted pentagonal bipyramidal geometry. In this description, the S(3) and C(31) atoms occupy axial positions so that the S(3)-Sn-C(31) angle is 166.19(12)°. The deviations of the S(1), S(2), S(4), S(5) and S(6) from the least-squares plane through them are 0.161(1), -0.296(1), 0.212(1), 0.042(1) and –0.181(1) Å, respectively with the Sn atom lying 0.3549(3) out of this plane in the direction of the

C(31) atom. The two dithiocarbamato ligands in the pentagonal plane form more symmetric Sn-S bonds than the dithiocarbamato ligand that spans equatorial and axial sites; the shortest Sn-S distance involves the sulfur atom in the axial position, *i.e.* S(3). The overall coordination geometry matches closely those found in related $RSn(S_2CNEt_2)_3$ systems [1], *i.e.* R = Ph [2, 3]; R = Me [4]; and R = $^n$Bu [5].

## Experimental

*Preparation:* The compound was prepared using a literature procedure [2]. Colourless crystals were obtained from the slow evaporation of a $CH_2Cl_2$/EtOH (1/1) mixture; m.p. 155-156 °C. $^1$H NMR: δ 7.79-8.40 m (H-*o*), 7.24-7.44 m (H-*m*, -*p*), 3.58 d ($CH_2$, 7.2 Hz), 2.27-2.41 m (CH), 0.92 d ($CH_3$). $^{13}$C NMR: δ 157.3, 130.8, 127.9, 128.2 (Cα-Cδ, phenyl), 202.4 (Cq), 63.1 ($CH_2$), 26.9 (CH), 20.2 ($CH_3$). $^{119}$Sn NMR: δ -827.2 ppm. IR (KBr disk) ν(C-N) 1475, ν(C-S) 991 cm$^{-1}$.

*Crystallography:* The C(3)-C(5) atoms of a butyl group were found to be disordered so that two positions (50% site occupancy) were found for each of C(3) and C(5). The atoms of this group were refined isotropically and hydrogen atoms were not included. The residual electron density peak (1.42 e Å$^{-3}$) was located in this region. An empirical absorption correction was applied [6].

**Table 1.** Crystallographic data for $[PhSn(S_2CN^iBu_2)_3]$

| | | | |
|---|---|---|---|
| Formula | $C_{33}H_{59}N_3S_6Sn$ | Formula weight | 808.9 |
| Crystal system | triclinic | Space group | *P*bar1 |
| *a*, Å | 12.556(6) | *b*, Å | 16.888(5) |
| *c*, Å | 10.381(2) | α, ° | 96.92(2) |
| β, ° | 106.92(2) | γ, ° | 78.00(2) |
| V, Å$^3$ | 2056(1) | *Z* | 2 |
| Crystal size, mm | 0.08 x 0.15 x 0.37 | Diffractometer | Rigaku AFC7R |
| Temperature, K | 173 | μ(Mo-Kα), cm$^{-1}$ | 9.51 |
| $D_{calcd}$, g cm$^{-3}$ | 1.307 | *F*(000) | 848 |
| $\theta_{max}$, ° | 27.5 | No. reflns meas., unique | 9849, 9422 |
| No. reflns with $I \geq 2\sigma(I)$ | 5845 | *R*, *wR* ($F^2$, obs. data) | 0.040, 0.098 |
| *R*, *wR* ($F^2$, all data) | 0.101, 0.118 | No. parameters | 382 |
| Weighting scheme | $w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 1.6732P]$ where $P - (F_o^2 + 2F_c^2)/3$ | | |
| GoF | 1.01 | ρ, e Å$^{-3}$ | 1.42 |
| Programs used | DIFABS [6], teXsan [7], DIRDIF [8], SHELXL97 [9], ORTEP [10] | | |
| Deposition no. | CCDC 162240 | | |

**Acknowledgements**

The Australian Research Council is thanked for support.

## References

[1] E.R.T. Tiekink, *Main Group Met. Chem.*, **15** (1992) 161; **16** (1993) 129.
[2] D. Dakternieks, H. Zhu, D. Masi and C. Mealli, *Inorg. Chim. Acta*, **211** (1993) 155.
[3] E. Kellö, V. Vrábel, I. Ska_áni and J. Hole_ek, *Acta Crystallogr.*, **C51** (1995) 408.
[4] J.S. Morris and E.O. Schlemper, *J. Cryst. Mol. Struct.*, **8** (1978) 295.
[5] J.S. Morris and E.O. Schlemper, *J. Cryst. Mol. Struct.*, **9** (1979) 1.
[6] N. Walker and D. Stuart, *Acta Crystallogr.*, A**39** (1983) 158.
[7] teXsan, Single Crystal Structure Analysis Software, Version 1.04 (1997), Molecular Structure Corporation, The Woodlands, TX, U.S.A.
[8] P.T. Beurskens, G. Admiraal, G. Beurskens, W.P. Bosman, S. García-Granda, R.O. Gould, J.M.M. Smits and C. Smykalla, The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands (1992).
[9] G.M. Sheldrick, SHELXL97, University of Göttingen, Germany (1997).
[10] C.K. Johnson, ORTEPII, Report ORNL-5138, Oak Ridge National Laboratory, TN, U.S.A. (1976).

**Received: April 20, 2001 - Accepted: April 27, 2001 -**
**Accepted in publishable format: May 2, 2001**