Crystal structure of [*p*-(2-hydroxy-5-methylphenylazo)benzoato]triethyltin, $C_{20}H_{26}N_2O_3Sn$

T. S. Basu Baul* and E. R. T. Tiekink

The University of Adelaide, Department of Chemistry, Adelaide, S.A. 5005, Australia

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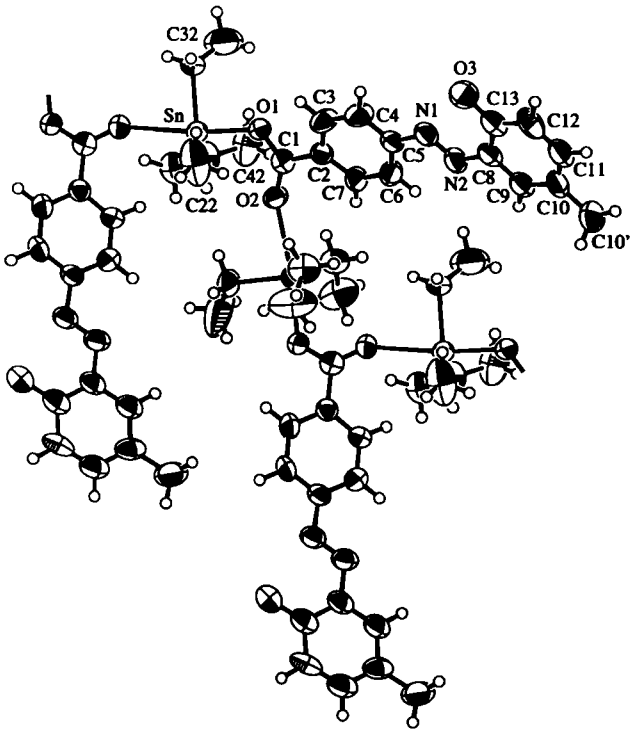

Source of material: Prepared by stirring Et_3SnBr with Na[*p*-(2-hydroxy-5-methylphenylazo)]benzoate in methanol solution for 2 h. Recrystallisation from a mixture of $CHCl_3$ and petroleum ether (303-313 K) (4/1) gave orange blocks in 71 % yield; mp 379 K-380 K.

The structure is polymeric owing to the presence of bidentate bridging carboxylate ligands which form disparate Sn–O(1) and Sn–O(2)' distances of 2.149(4) Å and 2.586(4) Å, respectively (symmetry operation: x, 0.5–y, −1.5+z). The Sn atom is thus fivefold-coordinated, exisiting in a distorted trigonal bipyramidal geometry with the Sn atom lying 0.1963(5) Å out of the C_3 plane in the direction of the O(1) atom. The axial O–Sn–O angle is 173.8(2)°. The structure reported here resembles closely that found for the Me_3Sn analogue (see ref. 1). Is is noted that the disparity in the Sn–O bond distances is greater in the Et_3Sn species (i.e. 0.437 Å cf. 0.358 Å). Both structures conform to a common motif, i.e. *trans*-C_3SnO_2, found for similar compounds (see ref. 2). Note, the H postion of the hydroxyl group was not located.

$C_{20}H_{26}N_2O_3Sn$, monoclinic, $P12_1/c1$ (No. 14), a =8.526(4) Å, b =24.715(5) Å, c =10.698(3) Å, β =107.95(3)°, V =2144.6 Å^3, Z =4, $R(F)$ =0.045, $R_w(F)$ =0.042.

Table 1. Parameters used for the X-ray data collection

Crystal:	orange block, size 0.08 x 0.19 x 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.11 cm^{-1}
Diffractometer:	Rigaku AFC6R
Scan mode:	ω/2θ
$T_{measurement}$:	293 K
$2\theta_{max}$:	55°
$N(hkl)_{unique}$:	5054
Criterion for F_o:	$F_o > 6\,\sigma(F_o)$
$N(param)_{refined}$:	235
Program:	teXsan

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	−0.24025	0.41739	0.49295	0.08974
H(4)	4*e*	−0.32893	0.48758	0.60263	0.10123
H(6)	4*e*	−0.09207	0.41870	0.95209	0.07921
H(7)	4*e*	−0.00395	0.34842	0.84464	0.07514
H(9)	4*e*	−0.20771	0.50462	1.20823	0.08033
H(10a)	4*e*	−0.16582	0.56543	1.42410	0.12589
H(10b)	4*e*	−0.32613	0.53037	1.40902	0.12589
H(10c)	4*e*	−0.33198	0.59402	1.42440	0.12589
H(11)	4*e*	−0.46956	0.63836	1.22696	0.09561
H(12)	4*e*	−0.54175	0.64926	1.00203	0.10296
H(21b)	4*e*	−0.31925	0.22767	0.22471	0.09427
H(21a)	4*e*	−0.25695	0.21269	0.37601	0.09427
H(22a)	4*e*	−0.36705	0.28796	0.42708	0.16287
H(22b)	4*e*	−0.50441	0.25739	0.31529	0.16287
H(22c)	4*e*	−0.41447	0.30855	0.27989	0.16287
H(31a)	4*e*	−0.15843	0.35627	0.16072	0.10251
H(31b)	4*e*	−0.01785	0.32585	0.12027	0.10251
H(32a)	4*e*	0.02298	0.40427	0.30211	0.29884
H(32b)	4*e*	0.16748	0.37221	0.27102	0.29884
H(32c)	4*e*	0.04748	0.40910	0.16165	0.29884
H(41b)	4*e*	0.23758	0.23102	0.37092	0.08590
H(41a)	4*e*	0.16941	0.21549	0.48820	0.08590
H(42a)	4*e*	0.25680	0.29822	0.58599	0.14285
H(42b)	4*e*	0.32610	0.31336	0.46908	0.14285
H(42c)	4*e*	0.40717	0.26556	0.56611	0.14285

* Permanent address: North Eastern Hill University, Regional Sophisticated Instrumentation Centre, Chemical Laboratory, Shillong 793003, India

Table 3. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	4e	–0.04445(6)	0.27974(2)	0.33108(4)	0.0484(3)	0.0505(3)	0.0474(2)	–0.0039(3)	0.0146(2)	0.0017(3)
O(1)	4e	–0.0615(6)	0.3396(2)	0.4721(4)	0.085(4)	0.048(3)	0.052(3)	0.000(2)	0.030(3)	0.001(3)
O(2)	4e	–0.0325(6)	0.2840(2)	0.6426(4)	0.084(3)	0.058(3)	0.055(3)	0.013(3)	0.029(2)	0.010(3)
O(3)	4e	–0.4633(8)	0.5877(2)	0.8332(6)	0.137(6)	0.067(4)	0.091(4)	0.026(3)	0.035(4)	0.009(4)
N(1)	4e	–0.2896(7)	0.5011(2)	0.8435(6)	0.080(5)	0.045(4)	0.076(4)	–0.002(3)	0.036(4)	–0.005(3)
N(2)	4e	–0.2525(8)	0.4982(2)	0.9667(6)	0.082(5)	0.050(4)	0.074(4)	–0.004(3)	0.033(4)	–0.012(3)
C(1)	4e	–0.0659(8)	0.3288(3)	0.5880(7)	0.049(4)	0.059(5)	0.062(4)	–0.001(4)	0.018(4)	–0.001(4)
C(2)	4e	–0.1161(8)	0.3754(3)	0.6576(6)	0.056(5)	0.047(4)	0.055(4)	–0.001(3)	0.023(4)	0.001(4)
C(3)	4e	–0.210(1)	0.4170(3)	0.5881(7)	0.101(7)	0.069(6)	0.057(4)	0.034(4)	0.028(4)	0.019(5)
C(4)	4e	–0.263(1)	0.4583(3)	0.6528(7)	0.130(8)	0.059(6)	0.071(5)	0.043(4)	0.040(5)	0.024(5)
C(5)	4e	–0.2234(9)	0.4582(3)	0.7860(7)	0.080(6)	0.040(4)	0.065(4)	–0.001(4)	0.033(4)	–0.001(4)
C(6)	4e	–0.1250(9)	0.4180(3)	0.8569(6)	0.078(6)	0.066(5)	0.051(4)	0.007(4)	0.016(4)	–0.003(5)
C(7)	4e	–0.0727(9)	0.3770(3)	0.7934(6)	0.080(6)	0.050(5)	0.057(4)	0.014(3)	0.019(4)	–0.003(4)
C(8)	4e	–0.3191(9)	0.5394(3)	1.0286(8)	0.073(6)	0.041(5)	0.084(5)	–0.006(4)	0.034(5)	–0.003(4)
C(9)	4e	–0.2775(9)	0.5344(3)	1.1649(8)	0.078(6)	0.046(5)	0.081(5)	–0.003(4)	0.030(5)	–0.005(4)
C(10)	4e	–0.334(1)	0.5708(3)	1.2393(8)	0.085(6)	0.056(6)	0.086(6)	–0.024(5)	0.043(5)	–0.024(5)
C(10')	4e	–0.285(1)	0.5645(4)	1.3878(8)	0.150(9)	0.086(7)	0.098(7)	–0.015(5)	0.065(7)	–0.028(6)
C(11)	4e	–0.430(1)	0.6123(3)	1.1758(9)	0.094(7)	0.054(6)	0.100(6)	–0.010(5)	0.042(6)	–0.016(5)
C(12)	4e	–0.473(1)	0.6188(3)	1.043(1)	0.093(7)	0.047(5)	0.123(8)	0.012(5)	0.041(6)	–0.018(5)
C(13)	4e	–0.420(1)	0.5820(3)	0.9649(8)	0.079(6)	0.048(5)	0.094(6)	–0.004(5)	0.032(5)	–0.004(4)
C(21)	4e	–0.2764(9)	0.2421(4)	0.3131(8)	0.041(4)	0.095(7)	0.097(6)	–0.015(5)	0.017(4)	–0.013(4)
C(22)	4e	–0.401(1)	0.2767(4)	0.336(1)	0.071(6)	0.16(1)	0.18(1)	–0.028(9)	0.052(7)	–0.081(7)
C(31)	4e	–0.047(1)	0.3424(3)	0.1926(7)	0.135(8)	0.060(6)	0.061(5)	–0.023(4)	0.029(5)	0.007(6)
C(32)	4e	0.056(2)	0.3849(6)	0.235(1)	0.39(2)	0.20(1)	0.088(8)	–0.194(9)	–0.03(1)	0.04(2)
C(41)	4e	0.1878(9)	0.2450(3)	0.4346(7)	0.053(5)	0.080(6)	0.076(5)	0.005(4)	0.011(4)	–0.016(4)
C(42)	4e	0.304(1)	0.2837(4)	0.5215(9)	0.067(6)	0.17(1)	0.105(7)	0.006(7)	0.008(5)	–0.037(7)

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References

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