# Alkenyl Derivatives of the Metals. Oxidative Addition to Intermediate Tin(II) Alkenyls; Isolation, and Crystal and Molecular Structure of n-Butyltris(triphenylethenyl)tin(IV) †

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Reaction of tin(II) chloride with Li(CPh=CPh$_2$) at −78 °C in diethyl ether–hexane–tetrahydrofuran affords a deep red solution whose colour fades on warming, and which we believe contains the (unstable) first dialkenyltin(II) species. The latter survives long enough at low temperatures to undergo intermolecular oxidative addition, and one such adduct leads ultimately to the formation of Sn(CPh=CPh$_2$)$_3$Bu$^n$, which has been fully characterised including a crystal and molecular structure study. The mechanism of formation of the final product has been examined and results are reported.

We have reported earlier on alkenyl derivatives of both main-group [1] and transition [2–4] elements with regard to their structure,[3] synthesis,[2,4] and properties. We now present details on the reactions of alkenylating agents with tin(II) chloride. Until relatively recently,[5] stable alkyls of bivalent tin were unknown. Earlier reports claiming such species have been recognised to relate to poly- or oligo-meric tin(IV) compounds.[6] Lappert and co-workers [5] subsequently obtained Sn[CH(SiMe$_3$)$_2$]$_2$, however, demonstrating that stable tin(II) alkyls can be obtained with bulky, kinetically stabilising ligands. An extensive chemistry has been reported for this compound, including facile oxidative addition with alkyl and aryl halides, and behaviour as both a Lewis acid and base.

We sought to prepare similarly stabilised alkenyl derivatives of tin(II) to extend the known chemistry of alkenyl compounds, and because no purely hydrocarbon derivatives of tin(II) are known with σ-bonding ligands. The only hydrocarbon derivatives reported are Sn(η-C$_5$H$_5$)$_2$ which has been shown to have a bent sandwich structure,[7] its methylcyclopentadienyl analogue, Sn(η-C$_5$H$_4$Me)$_2$,[8] and Sn(η-C$_5$Me$_5$)BF$_4$, which has a remarkable half-sandwich arrangement in the crystal.[9] Our earlier work with tin(IV) alkenyls included the ligands $^-$CMe=CMe$_2$, $^-$CPh=CMe$_2$, and $^-$CPh=CPh$_2$.[1] An unexpected observation in the course of this work was that Sn(CPh=CPh$_2$)$_4$ could not be obtained, even from Sn(CPh=CPh$_2$)$_3$Cl and Li(CPh=CPh$_2$) under prolonged reflux, a fact which we attribute to the steric requirement of the bulky alkenyl. [Sn(CPh=CMe$_2$)$_4$ was readily obtained.] [10] The triphenyl ligand was therefore the one chosen for study with SnCl$_2$.

## Results and Discussion

Addition of a tetrahydrofuran (thf) solution of tin(II) chloride to two equivalents of lithiotriphenylethene, prepared from bromotriphenylethene in ether and butyl-lithium in hexane,[1] gave a deep red solution at −78 °C. This colour discharged, even at −78 °C, to afford an orange suspension. After warming to room temperature, the only characterised product was a white crystalline solid which could be isolated in small yield. This was identified as n-butyltris(triphenylethenyl)tin(IV) (1) by a single-crystal *X*-ray structure determination, in agreement with elemental analysis, mass spectrometry, i.r. and n.m.r. spectroscopy.

It is thought that the initial red colour is due to the formation of the dialkenyl, Sn(CPh=CPh$_2$)$_2$ {*cf.* Sn[CH(SiMe$_3$)$_2$]$_2$, which is deep red}, and probable routes to the formation of the final product are shown in the Scheme. The order of the reactions of the intermediate tin(II) alkenyl with n-butyl bromide and the alkenyl-lithium is uncertain. Both reactions (i) and (ii) have precedents in the literature with analogous alkyl derivatives. Tetraorganotins were obtained by Gilman and Rosenberg [11] from the reaction of tin(II) chloride with lithium alkyls. Reaction with two equivalents of lithium alkyl gave deep red solutions, presumed to contain the unstable dialkyltin. Addition of a further equivalent of lithium reagent discharged the red colour and afforded SnR$_3$Li species, on which further chemistry was carried out. Thus reactions of type (ii) are established. Reactions of type (i) have only more recently been established in the chemistry of the more stable Sn[CH(SiMe$_3$)$_2$]$_2$ which adds oxidatively both alkyl and aryl halides.[5] Attempts at trapping the present dialkenyl by oxidative addition of methyl iodide at −78 °C failed, and addition of the lithiated olefin to the tin(II) chloride (rather than *vice versa*) did not lead to an isolable product. These two results together probably imply that step (ii) rather than step (i) leads to the isolated butyl compound. The low yield obtained when the lithium reagent is initially present in excess may be due to the removal of the intermediate tris-alkenyl lithium species in the equilibrium involving SnCl$_2$ shown in brackets in the Scheme. The species SnR$_2$ will be removed by other reactions, principally association.

The $^1$H n.m.r. spectrum of the compound shows the expected multiplet for the phenyl region together with higher field absorptions for the butyl group. In the $^{13}$C n.m.r. spectrum the four butyl carbons are readily identified (12—30 p.p.m. downfield from SiMe$_4$) and 13 of the expected 14 alkenyl carbons can be resolved at 20 MHz. Even at 75 MHz the final peak could not be resolved, and it is not possible to assign the olefinic carbon absorptions with certainty. The mass spectrum, obtained at 70 eV ($\approx 112 \times 10^{-19}$ J), showed interestingly a highest *m/e* value of 885 corresponding to Sn(CPh=CPh$_2$)$_3$, and no molecular ion. In the experiment in which methyl iodide was added at low temperature following Li(CPh=CPh$_2$), the product showed a peak at *m/e* 645, corresponding to Sn(CPh=CPh$_2$)$_2$Me, and indicating that an intermolecular oxidative addition of MeI to Sn(CPh=CPh$_2$)$_2$ had occurred.

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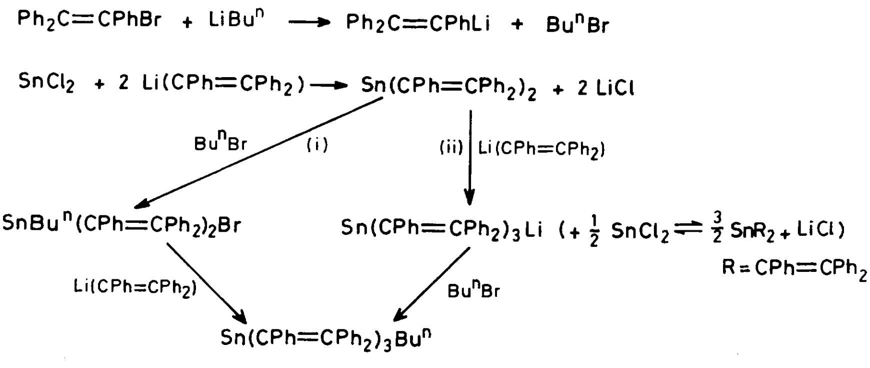
† *Supplementary data available* (No. SUP 23475, 13 pp.): observed and calculated structure factors, thermal parameters, hydrogen atom co-ordinates. See Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1981, Index issue.



**Scheme.**

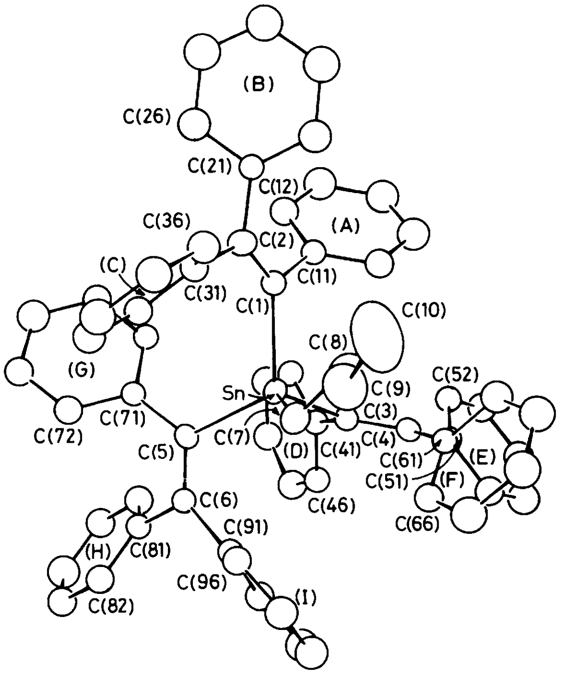


**Figure.** A single molecule of butyltris(triphenylethenyl)tin(IV), showing the numbering scheme. Phenyl ring carbon atoms are numbered C(*nm*) where *n* = 1 for ring A *etc.*, and *m* is the atom number within the ring

**Table 1.** Bond lengths (Å) and angles (°) around tin

| | |
|---|---|
| Sn–C(1) | 2.204(8) |
| Sn–C(3) | 2.187(7) |
| Sn–C(5) | 2.203(6) |
| Sn–C(7) | 2.168(8) |
| C(1)–Sn–C(3) | 105.6(3) |
| C(1)–Sn–C(5) | 113.6(4) |
| C(1)–Sn–C(7) | 103.7(3) |
| C(3)–Sn–C(5) | 99.8(3) |
| C(3)–Sn–C(7) | 119.4(3) |
| C(5)–Sn–C(7) | 114.9(3) |

**Table 2.** Short interligand contacts (Å)

| (*a*) C ··· C (<3.5 Å) | | (*b*) Sn ··· *o*-C(phenyl) | |
|---|---|---|---|
| C(7) ··· C(1) | 3.438(12) | (<3.6 Å) | |
| C(7) ··· C(31) | 3.473(12) | Sn ··· C(32) | 3.488(8) |
| C(7) ··· C(66) | 3.359(13) | Sn ··· C(96) | 3.502(9) |
| C(7) ··· C(96) | 3.259(12) | Sn ··· C(76) | 3.555(9) |
| C(3) ··· C(16) | 3.314(13) | | |
| C(3) ··· C(5) | 3.356(10) | (*c*) Sn ··· $C_\beta$ | |
| C(1) ··· C(76) | 3.480(11) | Sn ··· C(2) | 3.149(8) |
| C(71) ··· C(32) | 3.393(12) | Sn ··· C(4) | 3.179(8) |
| C(92) ··· C(46) | 3.434(13) | Sn ··· C(6) | 3.116(7) |

*Molecular Structure.*—In order to determine the structure of (1), an *X*-ray single-crystal diffraction study was carried out. The Figure shows a single molecule with the numbering scheme used. This analysis establishes the presence of the butyl group, suspected from the n.m.r. spectra, but not evident from the mass spectrum. As $Sn(CPh{=}CPh_2)_4$ cannot be synthesised, although the less crowded $Sn(CPh{=}CMe_2)_4$ can, compound (1) can be compared with the latter structure [10] which was initially examined for comparison with the analogous $Cr(CPh{=}CMe_2)_4$.[3,10]

It is immediately evident that the present structure is a sterically crowded one. In the $Sn(CPh{=}CMe_2)_4$ structure, all the Sn–C bond lengths are very similar, mean value 2.175(3) Å, and the bond angles show a distortion to approximate $S_4$ ($\bar{4}$) symmetry. In the present structure, both bond lengths and bond angles around the tin are much more irregular (Table 1). Most notably, the tin–butyl bond [Sn–C(7)] is shorter than the tin–alkenyl bonds and by more than 3σ when the contraction of the covalent radius of the carbon from 0.77 Å ($sp^3$) to 0.75 Å ($sp^2$) is taken into account. Notable also is that the distortion in the bond angles is not such as to crowd the butyl group directly; in fact there is one particularly small bond angle of 99.8(3)° between two alkenyl ligands [C(3)–Sn–C(5)]. The origin of the steric strain in the molecule is in the interactions between phenyl groups within each ligand. The ligand $-CPh{=}CMe_2$, with only α-phenyl groups, gives an unstrained tetra-alkenyl.* However, an examination of the short interligand C ··· C contact distances (Table 2) shows that the environment of C(7) (the α-carbon of the butyl ligand) is particularly crowded, with close contacts to atoms C(1), C(31), C(66), and C(96), that is, all three alkenyl ligands [see the Figure, which shows a projection of the molecule along a direction lying close to the Sn–C(7) vector]. The latter three contacts are to the *cis* β-phenyl groups of the three ligands, and must indirectly influence the orientations of all nine phenyl rings. There are also short contacts between the tin atom and three *ortho* carbon atoms of phenyl rings.

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* A referee has suggested that the Sn ··· $C_\beta$ distances [Sn ··· C(2) *etc.*] may show evidence of steric strain. The mean of these values (Table 2) is 3.148 Å, whereas the corresponding mean in $Sn(CPh{=}CMe_2)_4$ is 3.11 Å, so we feel that this is not a useful indicator.

**Table 3.** Bond lengths (Å) and angles (°) in the alkenyl and butyl ligands

(*a*) Alkenyl ligands

| Bond | Length (Å) |
|---|---|
| C(1)-C(2) | 1.351(11) |
| C(3)-C(4) | 1.324(10) |
| C(5)-C(6) | 1.339(12) |
| C(1)-C(11) | 1.504(11) |
| C(2)-C(21) | 1.513(11) |
| C(2)-C(31) | 1.488(11) |
| C(3)-C(41) | 1.528(10) |
| C(4)-C(51) | 1.507(11) |
| C(4)-C(61) | 1.529(12) |
| C(5)-C(71) | 1.483(13) |
| C(6)-C(81) | 1.504(9) |
| C(6)-C(91) | 1.518(11) |

Mean C-C(ethenyl) = 1.337(±12)
Mean C-C(phenyl) = 1.509(±15)
1.508(±15)

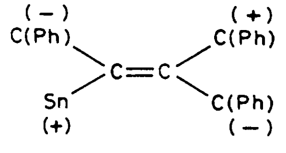
| Angle | (°) |
|---|---|
| Sn-C(1)-C(2) | 123.0(6) |
| Sn-C(1)-C(11) | 116.8(5) |
| C(11)-C(1)-C(2) | 119.8(7) |
| C(21)-C(2)-C(1) | 121.7(8) |
| C(31)-C(2)-C(1) | 125.6(7) |
| C(21)-C(2)-C(21) | 112.7(7) |
| Sn-C(3)-C(4) | 128.0(6) |
| Sn-C(3)-C(41) | 113.5(5) |
| C(41)-C(3)-C(4) | 118.5(7) |
| C(51)-C(4)-C(3) | 123.2(8) |
| C(61)-C(4)-C(3) | 122.5(7) |
| C(51)-C(4)-C(61) | 114.2(8) |
| Sn-C(5)-C(6) | 121.7(7) |
| Sn-C(5)-C(71) | 118.6(6) |
| C(71)-C(5)-C(6) | 120.1(6) |
| C(81)-C(6)-C(5) | 122.4(7) |
| C(91)-C(6)-C(5) | 123.9(7) |
| C(81)-C(6)-C(91) | 113.6(6) |

(*b*) Butyl ligand

| Bond | Length (Å) | Angle | (°) |
|---|---|---|---|
| C(7)-C(8) | 1.518(11) | Sn-C(7)-C(8) | 115.9(6) |
| C(8)-C(9) | 1.562(13) | C(7)-C(8)-C(9) | 109.5(8) |
| C(9)-C(10) | 1.313(17) | C(8)-C(9)-C(10) | 118.6(14) |

Examination of the dihedral angles between the phenyl rings and the appropriate alkenyl ' plane ' (Table 4) shows these angles (with the exception of ring F) to lie around a mean value of 58°. In the $Sn(CPh{=}CMe_2)_4$ structure this mean is 68°, whereas in the compounds $[M(\eta\text{-}C_5H_5)_2Cl(CPh{=}CMe_2)]$ (M = Ti, Zr, or Hf)[12] the angle is in each case close to 90°. The propeller-like arrangement of the phenyl groups around the double bond in the present structure is clearly determined by steric factors.

The remaining geometry of the alkenyl ligands is normal. Table 3 shows bond lengths and angles within the alkenyl groups and a summary of the results for the phenyl rings is presented in Table 4. Only the average bond lengths for each ring and the root-mean-square deviation from the least-squares plane are given, for brevity. These averages range from 1.375(12) to 1.389(9) Å. There is one anomalous value, that of 1.292(14) Å [C(63)-C(64)]. The bond distances C($sp^2$, alkenyl)-C($sp^2$, phenyl) and the metal-alkenyl bond distances themselves are also normal. However, it should be noted that the alkenyl ' planes ' are not strictly planar (Table 4). In every case the distortion is that expected for steric crowding around the alkenyl group, with *trans* ligands displaced in one direction from the plane, and the *cis* ligands in opposite directions (see below).



The geometry of the butyl group [Table 3(*b*)] is mainly of interest for the close contacts to the α-carbon C(7) (see above), resulting in a slight opening of the tetrahedral angle to 115.9(6)°. The terminal carbon atom, C(10), is sterically uncrowded and may be partially disordered, as shown by the apparent C(9)-C(10) bond length of 1.313(17) Å and the anomalously high thermal parameters for this atom (for details, see supplementary material).

**Table 4.** Dihedral angles between mean planes[a]

(*a*) Phenyl ring average bond lengths (Å) and planarity

| Ring | C-C(Å) | e.s.d.[b] | r.m.s.d.[c] |
|---|---|---|---|
| A | 1.384 | ±18 | 0.007 |
| B | 1.376 | ±31 | 0.012 |
| C | 1.384 | ±28 | 0.008 |
| D | 1.383 | ±24 | 0.004 |
| E | 1.382 | ±28 | 0.009 |
| F | 1.376 | ±50 | 0.0014 |
| G | 1.389 | ±09 | 0.008 |
| H | 1.375 | ±12 | 0.009 |
| I | 1.379 | ±34 | 0.020 |

(*b*) Angles (°) between mean planes

Plane 1: Sn, C(1), C(2), C(11), C(21), C(31); r.m.s.d. = 0.08
Plane 2: Sn, C(3), C(4), C(41), C(51), C(61); r.m.s.d. = 0.09
Plane 3: Sn, C(5), C(6), C(71), C(81), C(91); r.m.s.d. = 0.16

| | | | | | |
|---|---|---|---|---|---|
| 1-A | 60.3 | 2-D | 64.0 | 3-G | 49.5 |
| 1-B | 55.2 | 2-E | 50.7 | 3-H | 56.7 |
| 1-C | 56.2 | 2-F | 69.6 | 3-I | 56.8 |
| 1-2 | 85.6 | 2-3 | 73.3 | 3-1 | 83.0 |

[a] Planes A—I are defined as the mean planes through the nine phenyl rings. Planes 1—3 are the three alkenyl planes. [b] Estimated standard deviations are calculated from $[\Sigma(d_i - \bar{d})^2/N - 1]^{\frac{1}{2}}$, where $d_i$ is the mean distance, $\bar{d}$ the mean of the $N$ equivalent distances, and $N$ the number of observations. [c] Root mean square deviation of the carbon atoms from the least squares plane.

This is the first reported crystal structure of an organometallic molecule containing the ligand $-CPh{=}CPh_2$. The most closely related structures are our own studies of $M(CPh{=}CMe_2)_4$ (M = Sn or Cr),[3,10] and $[M'(\eta\text{-}C_5H_5)_2Cl(CPh{=}CMe_2)]$ (M′ = Zr or Hf).[12] The most closely related previous structures are those of a group of σ-butadienyl tin compounds.[13,14] In the case of Sn(*cis*,*cis*-CPh=CPh-CPh=

CPhX)PhMe$_2$ [X = Cl, (2); Br, (3)],[13] the geometry around the tin is less distorted than in (1), the only large angle being the C(1)(butadienyl)–Sn–C(5)(methyl) which is 117° in both. The Sn–C($sp^3$) distances are 2.180(10) and 2.154(10) Å for (2), and 2.188(15) and 2.165(13) Å for (3), which are comparable to the Sn–C($sp^3$) bond length in (1). The Sn–C($sp^2$) bond lengths in (2) and (3) are 2.145(8) (butadienyl) and 2.116(9) Å (phenyl), and 2.156(12) (butadienyl) and 2.141(13) Å (phenyl) respectively. For (2) these tin–butadienyl bond lengths are shorter by 3σ than any alkenyl–metal bond in (1). In (3), the difference is 2.5σ. In both cases they are significantly shorter. Steric crowding may be responsible for the lengthening of the alkenyl–tin bonds in (1). In Sn(*cis,cis*-CPh=CPh–CPh=CPhBr)Me$_2$Br (4),[14] the geometry around the tin is less regular and the C(1)(butadienyl)–Sn–C(5)(methyl) angle has opened up to 129.0(8)°. In this structure there is a short contact distance of 3.774(5) Å between the bromine atom and the tin, and the distortion is ascribed to this weak co-ordination of a fifth ligand. Tin–methyl bond lengths are close to the values for (2) and (3), though the tin–butadienyl bond, at 2.206(21) Å, is close to the Sn–C($sp^2$) distances found in (1).

## Experimental

All reactions were carried out using Schlenk tube techniques under an atmosphere of dry argon or nitrogen. Inert gases were purified by passage consecutively over activated BASF catalyst at 140 °C, 4A molecular sieve, and phosphorus pentoxide. Solvents were dried initially over sodium wire and distilled immediately before use from potassium benzophenone ketyl or a potassium mirror. Infrared spectra were recorded on a Perkin-Elmer 599 machine, $^1$H and $^{13}$C n.m.r. spectra on Bruker WP60, WP80, or 300 MHz machines, and mass spectra were recorded on an AEI MS9 instrument. Elemental analyses were performed by the Analytical Laboratory, University College, Dublin. Tin(II) chloride was used as received (B.D.H., anhydrous) and bromotriphenylethene was prepared by a literature method.[15]

*Isolation of n-Butyltris(triphenylethenyl)tin*(IV).—To bromotriphenylethene (9.64 g, 28.76 mmol) in ether (200 cm$^3$) cooled in an ethanol–dry-ice bath under argon was added dropwise n-butyl-lithium (14.4 cm$^3$ of a 1.975 mol dm$^{-3}$ solution, 28.44 mmol). After addition, the resultant lemon-yellow suspension was stirred (45 min) at low temperature and for 3 h at room temperature. The suspension was cooled, and a solution of tin(II) chloride (2.71 g, 14.31 mmol) in thf (75 cm$^3$) added dropwise. During the addition the mixture darkened to a deep red clear solution which finally lightened giving an orange suspension. After stirring at low temperature for 1 h, the suspension was allowed to warm to room temperature over 2 h. Volatiles were then removed under vacuum. Toluene (100 cm$^3$) was added to the residue and, after stirring (30 min), the mixture was filtered. Removal of the toluene under vacuum gave a yellow residue. Hexane (100 cm$^3$) was added; an aliquot (10 cm$^3$) did not react with an ether solution of methyl iodide. The remainder of the hexane suspension was filtered to afford a white solid which was recrystallised twice from hexane–toluene to afford white crystals of Sn(CPh=CPh$_2$)$_3$Bu$^n$ (0.301 g, 3.7%), m.p. 222 °C (Found: C, 81.5; H, 5.65%. $C_{64}H_{54}Sn$ requires C, 81.6; H, 5.8%). $^1$H N.m.r. ($C_6D_6$): δ 0.464 (s), 0.693 (br), 1.566 (s), 6.5—8.0 p.p.m. (m). $^{13}$C N.m.r. (CDCl$_3$): δ 13.515, 22.689, 27.109, 27.326, 124.917, 125.836, 127.192, 127.301, 128.660, 129.947, 130.231, 130.330, 144.064, 145.343, 145.999, 148.678, 153.085 p.p.m. I.r. (Nujol): 660vw, 670w, 700vs, 730m, 765m, 770m, 800m, 920w br, 963w, 1 030m, 1 074m, 1 150w br, 1 184vw, 1 572vw, 1 594w cm$^{-1}$.

*Attempted Trapping of* Sn(CPh=CPh$_2$)$_2$.—The above experiment was repeated with the following quantities: bromotriphenylethene (5.022 g, 14.98 mmol), n-butyl-lithium (7.4 cm$^3$ of 1.975 mol dm$^{-3}$ solution, 14.62 mmol), and tin(II) chloride (1.493 g, 7.87 mmol). When the deep red solution was obtained at low temperature and appeared just to be fading, an ether solution containing excess methyl iodide was added. Work up as above afforded reddish crystals of butyltris(triphenylethenyl)tin(IV) (0.136 g, 2.9%), m.p. 213—215 °C (Found: C, 81.4; H, 5.5%. Calc. for $C_{64}H_{54}Sn$: C, 81.6; H, 5.8%).

*Reversal of the order of addition.* In a further experiment the order of mixing the lithiated olefin and tin(II) chloride was reversed. No pure product could be isolated on work up.

*Crystal Data for* (1).—$C_{64}H_{54}Sn$, $M$ = 941.82, Orthorhombic, $a$ = 12.486(2), $b$ = 15.667(2), $c$ = 25.605(2) Å, $U$ = 5 009 Å$^3$, $D_m$ = 1.05 g cm$^{-3}$ (by flotation), $Z$ = 4, $D_c$ = 1.25 g cm$^{-3}$, $F$(000) = 1 952, space group $P2_12_12_1$, Mo-$K_\alpha$ radiation, λ = 0.710 69 Å, μ(Mo-$K_\alpha$) = 4.86 cm$^{-1}$. The compound crystallised as colourless rhombs. A crystal of approximate dimensions 0.2 × 0.2 × 0.3 mm was mounted up the 010 axis.

*Measurements.*—The space group and preliminary unit-cell dimensions were determined from Weissenberg photographs. Final values of the unit-cell dimensions and the intensities of 6 085 reflections in the range $2 \leqslant \theta \leqslant 27°$ were measured on a Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator and zirconium attenuator. A θ—2θ scan was employed for all intensities measured: if $\sigma/I < 0.5$ on the prescan, no final scan was carried out. The counting time was adjusted to obtain $\sigma/I \leqslant 0.02$ subject to a 120 s maximum. Two intensity and two orientation standards were checked periodically during data collection and found to show no significant fluctuations.

*Structural Analysis.*—Processing of the measured data yielded 3 259 independent reflections for which $I > 2.5\sigma(I)$. Absorption corrections were not applied (μ = 4.86 cm$^{-1}$ for Mo-$K_\alpha$). The position of the tin atom was found from a three-dimensional Patterson map, and also by the direct method using the program MULTAN (though no carbon atoms could be located this way). Subsequent difference syntheses revealed the positions of all non-hydrogen atoms. The tin atom position at approximately ¼, 0, 0.1 generated pseudo-symmetry when used as a phasing model (a false mirror plane) which made interpretation of initial difference syntheses and choice of a single image rather difficult.

The completed structure refined straightforwardly by the least-squares method. Isotropic refinement of the positional and vibrational parameters (with the phenyl rings constrained to refine as rigid groups) gave $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$ = 0.0514 and $R' = \{\Sigma(|F_o| - |F_c|)^2/\Sigma|F_o|^2\}^{\frac{1}{2}}$ = 0.0567 after six cycles of refinement for all non-hydrogen atoms. A further three cycles of refinement in which the tin, and the alkenyl and alkyl (but not the phenyl) carbon atoms were given anisotropic thermal parameters, and the phenyl ring constraints were removed, gave $R$ = 0.0461 and $R'$ = 0.0524. A further four cycles of refinement incorporating geometrically placed hydrogen atoms on the phenyl rings and butyl ligand gave the final converged values $R$ = 0.0364 and $R'$ = 0.0381.

**Table 5**. Fractional co-ordinates of atoms with standard deviations in parentheses

| Atom | *X/a* | *Y/b* | *Z/c* |
|---|---|---|---|
| Sn | −0.252 82(6) | −0.038 26(3) | −0.091 77(2) |
| C(1) | −0.093 5(6) | −0.053 5(5) | −0.127 7(3) |
| C(2) | −0.053 5(7) | −0.129 9(5) | −0.142 2(3) |
| C(3) | −0.285 2(5) | 0.098 9(4) | −0.092 6(3) |
| C(4) | −0.335 9(7) | 0.143 7(5) | −0.128 9(3) |
| C(5) | −0.254 1(10) | −0.061 3(4) | −0.006 88(24) |
| C(6) | −0.340 4(6) | −0.042 8(6) | 0.022 1(3) |
| C(7) | −0.353 9(7) | −0.123 2(5) | −0.137 0(3) |
| C(8) | −0.328 6(7) | −0.129 0(6) | −0.194 9(3) |
| C(9) | −0.391 9(11) | −0.205 0(8) | −0.219 5(4) |
| C(10) | −0.364 7(16) | −0.231 5(11) | −0.266 3(6) |
| C(11) | −0.033 8(6) | 0.027 0(5) | −0.141 0(3) |
| C(12) | 0.068 6(7) | 0.039 3(7) | −0.121 3(3) |
| C(13) | 0.126 3(9) | 0.114 1(7) | −0.134 8(4) |
| C(14) | 0.079 2(9) | 0.173 0(8) | −0.166 7(4) |
| C(15) | −0.021 2(8) | 0.160 4(7) | −0.186 9(4) |
| C(16) | −0.078 1(8) | 0.088 2(7) | −0.173 3(4) |
| C(21) | 0.047 1(7) | −0.137 1(5) | −0.174 9(3) |
| C(22) | 0.057 8(9) | −0.098 0(7) | −0.222 3(4) |
| C(23) | 0.152 0(10) | −0.107 1(7) | −0.251 3(5) |
| C(24) | 0.233 7(11) | −0.157 3(6) | −0.233 4(4) |
| C(25) | 0.223 5(10) | −0.198 4(7) | −0.188 5(4) |
| C(26) | 0.129 9(8) | −0.189 4(6) | −0.158 0(4) |
| C(31) | −0.101 0(7) | −0.214 6(5) | −0.129 5(3) |
| C(32) | −0.125 5(7) | −0.235 3(5) | −0.078 3(3) |
| C(33) | −0.170 8(8) | −0.314 4(7) | −0.065 4(4) |
| C(34) | −0.188 9(8) | −0.371 3(7) | −0.105 7(4) |
| C(35) | −0.167 7(8) | −0.352 8(7) | −0.155 4(4) |
| C(36) | −0.120 4(7) | −0.273 7(6) | −0.168 8(4) |
| C(41) | −0.241 2(9) | 0.145 9(4) | −0.044 92(25) |
| C(42) | −0.133 4(7) | 0.150 6(6) | −0.033 4(3) |
| C(43) | −0.100 2(9) | 0.193 8(6) | 0.011 3(4) |
| C(44) | −0.174 0(9) | 0.231 6(7) | 0.042 7(4) |
| C(45) | −0.280 4(8) | 0.229 9(6) | 0.033 0(4) |
| C(46) | −0.315 3(7) | 0.185 3(5) | −0.011 9(3) |
| C(51) | −0.341 9(7) | 0.239 8(5) | −0.128 4(3) |
| C(52) | −0.252 7(11) | 0.288 7(5) | −0.123 1(3) |
| C(53) | −0.257 1(11) | 0.377 2(5) | −0.120 8(3) |
| C(54) | −0.353 1(9) | 0.416 9(7) | −0.125 2(4) |
| C(55) | −0.445 5(10) | 0.370 7(7) | −0.131 6(4) |
| C(56) | −0.440 6(8) | 0.279 4(6) | −0.134 2(4) |
| C(61) | −0.401 5(7) | 0.101 6(6) | −0.172 2(4) |
| C(62) | −0.372 8(9) | 0.115 5(6) | −0.224 0(4) |
| C(63) | −0.443 3(9) | 0.081 3(7) | −0.262 5(4) |
| C(64) | −0.527 9(9) | 0.039 6(8) | −0.248 7(4) |
| C(65) | −0.559 2(10) | 0.024 5(8) | −0.198 8(4) |
| C(66) | −0.491 8(7) | 0.057 5(6) | −0.158 6(4) |
| C(71) | −0.153 6(7) | −0.089 5(5) | 0.018 7(3) |
| C(72) | −0.157 1(7) | −0.156 4(5) | 0.054 8(3) |
| C(73) | −0.065 0(7) | −0.184 7(6) | 0.079 0(4) |
| C(74) | 0.033 6(9) | −0.148 0(7) | 0.067 9(4) |
| C(75) | 0.038 5(9) | −0.082 9(6) | 0.031 1(4) |
| C(76) | −0.053 3(7) | −0.054 2(6) | 0.006 8(3) |
| C(81) | −0.334 6(6) | −0.031 2(5) | 0.080 3(3) |
| C(82) | −0.404 6(7) | −0.073 0(6) | 0.113 6(3) |
| C(83) | −0.399 6(8) | −0 060 2(7) | 0.167 2(4) |
| C(84) | −0.325 0(9) | −0.004 5(7) | 0.186 6(4) |
| C(85) | −0.256 6(11) | 0.038 2(6) | 0.154 6(3) |
| C(86) | −0.262 8(8) | 0.025 8(5) | 0.100 8(3) |
| C(91) | −0.451 4(7) | −0.028 0(6) | −0.000 4(3) |
| C(92) | −0.514 3(7) | 0.040 9(7) | 0.015 7(3) |
| C(93) | −0.617 4(8) | 0.050 3(7) | −0.004 8(4) |
| C(94) | −0.656 4(9) | −0.008 1(7) | −0.037 7(4) |
| C(95) | −0.600 9(8) | −0.076 9(7) | −0.052 5(4) |
| C(96) | −0.497 0(7) | −0.090 1(6) | −0.032 5(3) |

(When this stage of refinement was repeated with the signs of all co-ordinates reversed, the final converged values were $R = 0.0388$ and $R' = 0.0404$. The original choice of enantiomorph was therefore taken to be the correct one.) In this refinement all the hydrogen atoms attached to phenyl rings were given a common thermal parameter, but those on the butyl ligand were given individual thermal parameters (since these were expected to refine to higher values). In the final cycle of least-squares refinement, the only significant parameter shifts were those associated with the terminal carbon atom of the butyl group. A final difference-Fourier synthesis showed a maximum peak height of 0.46 e $Å^{-3}$ and a minimum of 0.36 e $Å^{-3}$ (in the region of the tin atom). The minimum peak height of a carbon atom in the final Fourier syntheses was 2.8 e $Å^{-3}$. Unit weights were used throughout refinement.

The programs SHELX, *X*-RAY 76, and XANADU were used for all calculations using the DEC-2060 computer in Trinity College, Dublin, and the ICL-2972 in the University of Edinburgh. Table 1 gives bond lengths and angles around the tin atom, Table 2 lists short interligand contacts, Table 4 the dihedral angles between mean planes, Table 3 is a summary of bond lengths and angles within the ligand, and Table 5 gives the fractional co-ordinates for all non-hydrogen atoms. Literature values for atomic scattering factors were used.[16]

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