Inorganica Chimica Acta, 133 (1987) 289–294

The Synthesis and Crystal Structures of Triphenyl(1,2,3-trimethylallyl)tin and Triphenyl(1,1,2-trimethylallyl)tin

CRAIG M. VAN ZYL, JEFFREY L. McKEEBY, PAUL C. VAN DORT, ERLUND J. LARSON, MICHAEL E. SILVER*

Department of Chemistry, Hope College, Holland, Mich. 49423, U.S.A.

and JOHN C. HUFFMAN

Department of Chemistry, Indiana University, Bloomington, Ind. 47405, U.S.A.

(Received February 10, 1987)

Abstract

In an attempt to isolate new allylating reagents, two new tin compounds, triphenyl(1,2,3-trimethylallyl)tin (**3**) and triphenyl(1,1,2-trimethylallyl)tin (**4**) have been prepared and their crystal structures determined. Both compounds are examples of σ-bound allyl complexes. However, the infrared spectra of both compounds do not show the absorptions characteristic of other σ-bound allyl complexes. The chemistry of these new compounds also differs significantly from that of similar triphenyltin(allyl) reagents.

Introduction

Many transition metal allyl complexes are most easily prepared by reacting a transition metal halide with a Grignard or lithium allylating reagent [1–7]. The synthesis of the lithium allylating reagents most often involves the reaction of the appropriate triphenyltin(allyl) with phenyl lithium [8]. This approach, however, has been restricted to the preparation of unmethylated and monomethylated lithium allyls. In our attempts to produce highly methylated allylating reagents we have prepared two new tin allyl compounds, triphenyl(1,2,3-trimethylallyl)tin (**3**) and triphenyl(1,1,2-trimethylallyl)tin (**4**) and have explored the effects of allyl trimethylation on the chemical and spectral properties of these compounds.

Experimental

Physical Measurements

(a) Nuclear magnetic resonance spectra. Proton chemical shifts were measured at ambient probe temperature with a Varian FT-80A 80-MHz spectrometer.

(b) Infrared spectra. Infrared spectra of the tin compounds were recorded in the region 4000–300 cm^{-1} with a Nicolet 20-DX FT spectrophotometer equipped with a CsI beam splitter. The compounds were studied as mineral oil and Halocarbon 25-5S grease (polychlorotrifluoroethylene oils thickened with silica gel, Halocarbon Products Corp., N.J.) mulls between CsI plates. The estimated uncertainty in reported frequencies is ±2 cm^{-1}.

(c) Mass spectra. Mass spectra were measured on a Hewlett Packard 5995A mass spectrometer (EI at 70 eV) with the use of both GC and direct insertion probe inlet systems.

(d) Crystrallography. Pertinent data for the structures of compounds **3** and **4** are in Table I. Colorless crystals were obtained from hexane as described in 'Synthesis'. The crystals were mounted on a Picker computer controlled four-circle diffractometer equipped with a Furnas Monochromator (HOG crystal), and cooled by a gaseous nitrogen cooling system. A systematic search of a limited hemisphere of reciprocal space located a set of diffraction maxima with no apparent symmetry or absences for both compounds, indicating a triclinic space group. Subsequent statistical tests suggested the centrosymmetric space group $P\bar{1}$, and this choice was confirmed by the subsequent solution and refinement of the structures. Orientation matrices and accurate unit cell dimensions were determined at low temperature from a least-squares fit of 32 reflections ($20° < 2\theta < 30°$) for both **3** and **4**. Intensity data were collected using the θ–2θ scan method; 4 standard reflections, monitored every 300 reflection measurements, showed only statistical fluctuations for both compounds. Based on the cited dimensions of the crystals and the small linear absorption coefficients, absorption corrections were judged unnecessary. The intensities were then corrected for Lorentz and polarization factors and scaled to give the numbers of independent F_{hkl} values for $I > 3\sigma(I)$ indicated in Table I.

Both structures were solved by a combination of direct methods (MULTAN78) and Fourier techniques.

---

*Author to whom correspondence should be addressed.

0020-1693/87/$3.50

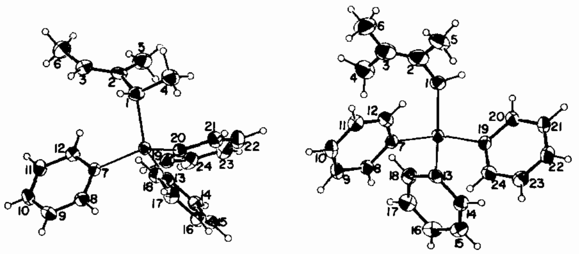
TABLE I. Crystal Data and Experimental Details for ϕ_3Sn(1,2,3-trimethylallyl) (3) and ϕ_3Sn(1,1,2-trimethylallyl)tin (4)

	3	4
Formula	$C_{24}H_{26}Sn$	$C_{24}H_{26}Sn$
Formula weight	433.16	433.16
a (Å)	9.727(4)	10.903(2)
b (Å)	12.015(4)	11.700(2)
c (Å)	9.958(3)	8.909(1)
α (°)	103.05(1)	91.29(1)
β (°)	95.15(2)	106.68(1)
γ (°)	104.97(2)	108.71(1)
V (Å^3)	1027.57	1022.83
Z	2	2
D_{calc} (g/cm^3)	1.401 (−162 °C)	1.406 (−155 °C)
Space group	$P\bar{1}$	$P\bar{1}$
Crystal dimensions	0.12 X 0.12 X 0.15 mm	0.18 X 0.18 X 0.20 mm
Radiation	Mo Kα (λ = 0.71069 Å)	Mo Kα (λ = 0.71069 Å)
μ (cm^{-1})	12.491	12.549
Receiving aperture	3.0 X 4.0 mm; 22.5 cm from xtal	3.0 X 4.0 mm 22.5 cm from xtal
Take off angle	2.0°	2.0°
Scan speed (° 2θ/min)	4.0	4.0
Background counts	8 s at each end	8 s at each end
2θ limits (°)	6.0 to 50.0	6.0 to 45.0
Data collected	$+h, \pm k, \pm l$	$+h, \pm k, \pm l$
Unique data	3617	3615
Unique data with $F_o^2 > 3\sigma(F_o^2)$	3252	3377
$R(F)$	0.0264	0.0292
$R_w(F)$	0.0284	0.0336

All atoms, including hydrogens, were located and refined. Full-matrix least-squares refinements included anisotropic thermal parameters for non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms; refinements converged to values for the conventional R indicies shown in Table I. A final difference Fourier for compound 3 contained two peaks of density 1.1 e/Å^3 near the Sn atom, and all other peaks were less than 0.5 e/Å^3. A final difference Fourier synthesis for compound 4 was essentially featureless, with the largest peak being 0.20 e/Å^3. The weighting scheme used in the final calculations was of the form $w = 1/\sigma_F^2$. Scattering factors for the atoms were taken from the International Tables [9]. The scattering factor for the Sn atom was corrected for the real and imaginary parts of anomalous dispersion using values from the International Tables [9]. All computations were carried out on a CDC Cyber 170-855 computer using programs described elsewhere [10]. The final positional and thermal parameters of the non-hydrogen atoms are listed in Table II, the atom-numbering scheme being shown in Fig. 1.

Synthesis of Compounds

The bromo-allyls (compounds **1** and **2**) were stored over $MgSO_4$ under argon in a freezer until used. Tetrahydrofuran used in the Grignard preparations of the ϕ_3Sn(trimethylallyl) (ϕ = phenyl) compounds was distilled from sodium/benzophenone. Triphenyltinchloride was purchased from Aldrich Chemical Company and used without further purification. The ϕ_3Sn(trimethylallyl) compounds were air stable and were recrystallized from reagent grade hexanes in open beakers placed in a freezer (−12 °C).



3 4

Fig. 1. ORTEP drawings of ϕ_3(1,2,3-trimethylallyl)tin (3) and ϕ_3(1,1,2-trimethylallyl)tin (4) showing atom labeling schemes.

4-Bromo-3-methyl-2-pentene(1,2,3-trimethylallyl-bromide (1)

Preparation of **1** was accomplished via hydrobromination of 4-hydroxy-3-methyl-2-pentene.

TABLE II. Fractional Atomic Coordinates (×10^4) and Thermal Parameters for Triphenyl(1,2,3-trimethylallyl)tin (3) and Triphenyl(1,1,2-trimethylallyl)tin (4)

3					4				
Atom	*x/a*	*y/b*	*z/c*	β (Å^2)	Atom	*x/a*	*y/b*	*z/c*	β (Å^2)
Sn	6582.4(2)	1424.6(2)	3143.2(3)	1.9	Sn	8505.9(2)	1414.4(2)	4003.5(3)	2.0
C(1)	7630(4)	2534(3)	5453(4)	2.8	C(1)	9316(4)	1348(4)	6511(4)	2.8
C(2)	7197(4)	3713(3)	5992(4)	2.7	C(2)	8380(5)	1563(5)	7362(5)	4.5
C(3)	6169(4)	3777(4)	6771(4)	3.0	C(3)	8515(6)	2613(5)	7940(5)	4.9
C(5)	7938(6)	4750(4)	5561(6)	3.9	C(5)	7163(5)	424(4)	7382(6)	4.0
C(6)	5542(6)	4824(5)	7340(6)	3.9	C(6)	7552(8)	2832(7)	8782(7)	5.5
C(7)	4287(3)	1017(3)	2986(3)	2.0	C(7)	7289(3)	2563(3)	3667(4)	2.2
C(8)	3373(4)	−230(3)	2425(4)	2.6	C(8)	7626(4)	3606(3)	2929(4)	2.4
C(9)	1887(4)	−505(4)	2369(4)	3.2	C(9)	6857(4)	4363(3)	2720(4)	2.7
C(10)	1283(4)	454(4)	2863(4)	3.1	C(10)	5728(4)	4088(3)	3239(5)	2.9
C(11)	2165(4)	1687(4)	3417(4)	2.6	C(11)	5373(4)	3060(3)	3977(5)	2.8
C(12)	3643(4)	1967(3)	3483(4)	2.2	C(12)	6153(4)	2305(3)	4186(4)	2.6
C(13)	7102(3)	−299(3)	2350(4)	2.0	C(13)	78(3)	2167(3)	2946(4)	2.3
C(14)	7422(4)	−753(3)	950(4)	2.4	C(14)	132(4)	1555(3)	1619(4)	2.7
C(15)	7768(4)	−1861(3)	442(4)	2.7	C(15)	1140(4)	2059(4)	929(5)	3.1
C(16)	7792(4)	−2532(3)	1312(4)	2.8	C(16)	2110(4)	3282(4)	1550(5)	3.3
C(17)	7474(4)	−2096(3)	2695(4)	2.9	C(17)	2077(4)	3813(4)	2865(5)	3.3
C(18)	7139(4)	−984(3)	3211(4)	2.6	C(18)	1069(4)	3305(3)	3558(4)	2.8
C(19)	7268(4)	2433(3)	1803(4)	2.2	C(19)	7296(3)	−380(3)	2825(4)	2.3
C(20)	8707(4)	2745(3)	1637(4)	2.9	C(20)	7311(4)	−1396(3)	3610(4)	2.7
C(21)	9158(5)	3446(4)	815(5)	3.4	C(21)	6580(4)	−2565(3)	2833(5)	3.0
C(22)	8183(5)	3837(4)	149(5)	3.4	C(22)	5828(4)	−2740(4)	1257(5)	3.1
C(23)	6745(5)	3523(4)	296(4)	3.2	C(23)	5796(4)	−1749(4)	452(5)	3.2
C(24)	6297(4)	2834(3)	1119(4)	2.6	C(24)	6519(4)	−577(4)	1233(4)	2.8

Vigorous shaking in a separatory funnel of 38.3 g of 4-hydroxy-3-methyl-2-pentene (0.38 mol) with excess (130 ml) HBr (47%) for 30 min yielded the crude yellow liquid **1**, which was dried over anhydrous $MgSO_4$ and purified by fractional distillation (boiling point (b.p.) 40–42 °C at 10 torr) to yield **1** as a colorless liquid (21.4 g (34.3%) recovered), revealed by GC/MS to be a mixture of both E and Z isomers. Aqueous washes before distillation converted **1** back to the alcohol and were thus avoided. *Anal.* Calc. for $C_6H_{10}Br$: C, 44.20; H, 6.80; Br, 49.00. Found: C, 44.35; H, 6.66; Br, 49.48%. Mass spectrum *m/e* (rel. intensity) 164, 162 (M^+, 1:1, 1.1), 67(100). ^1H NMR ($CDCl_3$/TMS-2%) δ (CH_3) m, 1.59, 1.65, 1.72, 1.74, 1.80; (CH) q, 5.64; (CH) q, 5.23; (CH) q, 4.47.

4-Bromo-2,3-dimethyl-2-butene(1,1,2-trimethylallylbromide) (2)

Preparation of **2** was similar to that of compound **1**. Treatment of 32.8 g (0.33 mol) of 4-hydroxy-2,3-dimethyl-2-butene with 110 ml HBr (47%) yielded the crude yellow liquid **2**, which was washed three times with water, dried over anhydrous $MgSO_4$, and purified by fractional distillation (b.p. 42–52 °C at 20 torr) to yield **2** as a colorless liquid (21.8 g (40.8%) recovered). The yield increased substantially if the reaction was run on a smaller scale. *Anal.* Calc. for $C_6H_{10}Br$: C, 44.20; H, 6.80; Br, 49.00. Found: C, 44.86; H, 7.19; Br, 48.42%. Mass spectrum *m/e* (rel. intensity) 164, 162 (M^+, 1:1, 9.5), 67(100). ^1H NMR ($CDCl_3$/TMS-2%) δ (CH_3) 1.76(6H); 1.70(3H); (CH_2) 4.05.

Triphenyl(1,2,3-trimethylallyl)tin (3)

Magnesium turnings (3.90 g, 0.160 mol) were placed in a three neck flask equipped with a gas inlet, water condenser, and an addition funnel. Tetrahydrofuran (70 ml) was added and the reaction was initiated by addition of a small amount of 1,2,3-trimethylallylbromide (~3 ml). The solution was heated to reflux under a nitrogen atmosphere. A solution consisting of triphenyltinchloride (15.1 g, 0.039 mol) in THF (50 ml) and the remainder of the 1,2,3-trimethylallylbromide (total of 10.0 g, 0.156 mol) was added to the reaction flask over 2.5 h. After 22 h of additional stirring with reflux, the mixture was hydrolyzed with saturated ammonium chloride solution and the organic layer was isolated and combined with the ether washings of the aqueous layer. The ether and THF were removed by rotary evaporation and the coupled bis-allyls were distilled off under reduced pressure (68–70 °C at 12 torr). The remaining residue was dissolved in ether (50 ml) and washed with 20 ml of a methanol-water (1:1) solution of KF (10%) to precipitate any unreacted triphenyltinchloride as triphenyltinfluoride. The ether layer was then dried with anhydrous $MgSO_4$ and the ether removed by rotary evaporation. Hexane (30 ml) was then added and successive crystallizations were done (−12 °C), yielding 6.13 g (36%) of white, crystalline (melting point (m.p.) 60–62 °C) triphenyl(1,2,3-trimethylallyl)tin. *Anal.* Calc. for $C_{24}H_{26}Sn$: C, 66.55; H, 6.05; Sn, 27.40. Found: C, 66.44; H, 5.98; Sn, 27.45%. ^1H NMR ($CDCl_3$/TMS-2%) δ (CH_3) 1.62, 1.55, 1.45; (CH) q, 5.23; (CH) q, 2.85; (phenyl) m, 7.36. Mass spectrum *m/e* (rel. intensity), 434 (M^+, 0.9), 351(100). IR (cm^{-1}, Nujol mull): 1090m, 1030w, 1010w, 840w, 740s, 720s, 675w, 460s; (cm^{-1}, Halocarbon grease mull): 1477s, 1425s. Scale-up of this reaction dramatically reduced the yield, and the first crop of solid upon successive crystallizations yielded triphenyl(4-hydroxybutyl)tin [white, crystalline solid, m.p. 66–68 °C. *Anal.* Calc. for $C_{22}H_{24}OSn$: C, 62.95; H, 5.72; Sn, 28.05. Found: C, 62.39; H, 5.71; Sn, 27.69%], resulting from a ring opening reaction of the THF solvent [11].

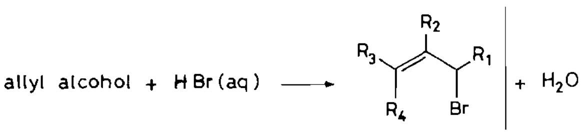
Triphenyl(1,1,2-trimethylallyl)tin (4)

Compound **4** was prepared using the same scale and method employed for compound **3**, yielding 9.96 g (59%) of white, crystalline (m.p. 61–64 °C) triphenyl(1,1,2-trimethylallyl)tin. *Anal.* Calc. for $C_{24}H_{26}Sn$: C, 66.55; H, 6.05; Sn, 27.40. Found: C, 66.42; H, 6.04; Sn, 27.32%. ^1H NMR ($CDCl_3$/TMS-2%) δ (CH_3) 1.57, 1.53; (CH_2) s, 2.41; (phenyl) m, 7.34. Mass spectrum *m/e* (rel. intensity) 434 (M^+, 5.8), 351(100). IR (cm^{-1}, Nujol mull): 1074s, 1022m, 997m, 728s, 698s, 453s, 441s, 327w; (cm^{-1}, Halocarbon grease mull): 1477s, 1425s.

Results and Discussion

(1) Preparation and Properties of the Allyl Bromides

1,2,3-Trimethylallylbromide (**1**) and 1,1,2-trimethylallylbromide (**2**) were both prepared in moderate yield via reaction of the corresponding allyl alcohol with concentrated aqueous HBr, and were purified by reduced pressure distillation. Compound **1** had not been reported previously. On distillation, both compounds were colorless. However, even when stored in the dark over $MgSO_4$ and under argon at −12 °C, both compounds darkened considerably, and a more dense liquid phase collected on the bottom of the flask. Nevertheless, NMR and GC/MS spectra of these compounds both freshly distilled and after appreciable darkening were identical, and the darkened material could be used without redistillation. Note that 1,2,3-trimethylallylbromide (**1**), a secondary allyl bromide, readily converted back to the allyl alcohol on aqueous washing, whereas the primary allyl bromide 1,1,2-trimethylallylbromide (**2**), did not.

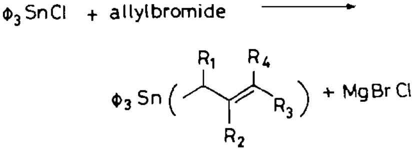


1,2

1 R_1, R_2, R_3 = Me; R_4 = H
2 R_1 = H; R_2, R_3, R_4 = Me

(2) Preparation and IR Spectra of the Triphenyltin Allyl Compounds

Triphenyl(1,2,3-trimethylallyl)tin (**3**) and triphenyl(1,1,2-trimethylallyl)tin (**4**) were prepared in a fashion analogous to the unmethylated allyl compound triphenyl(allyl)tin [8]. In the preparation of compound **3**, a competing side reaction [11] involving opening of the THF ring to produce triphenyl(4-hydroxybutyl)tin occurred whenever the reaction was scaled up appreciably beyond that described in 'Experimental' lowering the yield. This was never observed during the preparation of **4**; however, the yield also decreased substantially on scale up. We were able to obtain THF ring opening by simply running the reaction without allylhalide, demonstrating that the lack of this side reaction in the formation of **4** is simply a kinetic effect. Evidently, the primary allylbromide **2** reacts more quickly with magnesium than does triphenyltinchloride. The secondary allylbromide **1** reacts more slowly and the competitive reaction of triphenyltinchloride with the magnesium, followed by reaction with the solvent, becomes important.



3,4

3 R_1, R_2, R_3 = Me; R_4 = H
4 R_1 = H; R_2, R_3, R_4 = Me

Although compounds **3** and **4** are σ-bound allyl complexes (*vide infra*), they do not show the allylic C=C absorption in the region from 1650 to 1580 cm^{-1} expected for a σ-bound allyl [2]. This differs considerably from other allyl complexes. For example, the closely related unmethylated ϕ_3Sn(allyl) [12] exhibits a C=C stretching vibration at 1623 cm^{-1}. The frequency is approximately the same for the series $Bu_{3-n}(allyl)SnCl_n$ (n = 0–3) [13–15], as well as for other allylsilanes, allylgermanes, and allylstannanes [16, 17]. The spectrum of diallyldicyclopentadienylzirconium [2] shows strong absorptions at 1589 and 1533 cm^{-1}, interpreted as one σ and one

π-bound allyl. Even monomethylated σ-bound allyl compounds have absorptions in this region. For example, the spectrum of (monoethylallyl)dicyclopentadienylzirconium chloride [2] shows a band at 1606 cm^{-1}, indicative of its σ-bound allyl. Evidently, upon trimethylation, the allylic C=C stretch is too weak to be seen. Infrared spectra of compounds **3** and **4**, recorded as Halocarbon mulls in the region from 1400 to 1700 cm^{-1}, showed only two strong bands at 1477 and 1425 cm^{-1}, identical to the C=C stretching bands of the phenyl rings in ϕ_3SnCl [18].

(3) Attempts to Prepare Trimethylated Lithium Allyls

Lithium allyl and lithium monomethyl allyl have been prepared previously via the reaction shown below [8]. These reactions proceed instantaneously

$$\phi_3\mathrm{Sn(allyl)} + \phi\mathrm{Li} \xrightarrow{\mathrm{Et_2O}} \phi_4\mathrm{Sn} + \mathrm{Li(allyl)}$$

and with quantitative yield, precipitating a stoichiometric amount of ϕ_4Sn [8]. This does not occur when the trimethylallyl compounds **3** and **4** are used. These reactions proceed slowly and precipitate only ~40% of the expected amount of ϕ_4Sn, with no additional precipitation occurring after 24 h. Analysis of the reaction mixture showed small amounts of what appeared to be the lithium trimethylallyls along with significant quantities of reactants still present. Further attempts to increase the yield by varying the reaction conditions have so far proved unsuccessful. Thus, while this method may produce trimethylated lithium allyls, low yields and contamination by unreacted ϕLi and ϕ_3Sn(trimethylallyl) limit the usefulness of this approach to the preparation of lesser methylated lithium allyls.

TABLE III. Selected Structural Parameters[a] for Triphenyl(1,2,3-trimethylallyl)tin (3) and triphenyl(1,1,2-trimethylallyl)tin (4)

	3	4
Bond lengths (Å)		
Sn–C(1)	2.186(4)	2.167(4)
Sn–C(7)	2.137(3)	2.143(3)
Sn–C(13)	2.146(3)	2.144(3)
Sn–C(19)	2.145(3)	2.140(3)
C(1)–C(2)	1.508(5)	1.510(6)
C(1)–C(4)	1.533(5)	
C(2)–C(3)	1.320(5)	1.268(7)
C(2)–C(5)	1.494(6)	1.555(7)
C(3)–C(4)		1.533(7)
C(3)–C(6)	1.490(6)	1.531(8)
Bond angle (°)		
C(1)–Sn–C(19)	111.27(14)	109.79(15)
C(1)–Sn–C(7)	107.89(13)	108.87(13)
C(1)–Sn–C(13)	110.39(13)	112.41(14)
C(7)–Sn–C(19)	109.28(12)	110.14(13)
C(7)–Sn–C(13)	109.46(12)	107.09(13)
C(13)–Sn–C(19)	108.53(12)	108.51(13)
Sn–C(1)–C(2)	109.06(23)	110.11(25)
Sn–C(1)–C(4)	111.54(26)	
C(2)–C(1)–C(4)	115.2(3)	
C(1)–C(2)–C(3)	119.8(4)	122.7(5)
C(1)–C(2)–C(5)	117.2(3)	115.8(4)
C(3)–C(2)–C(5)	123.0(4)	121.3(5)
C(2)–C(3)–C(4)		119.7(5)
C(2)–C(3)–C(6)	127.9(4)	122.7(6)
C(4)–C(3)–C(6)		117.6(5)
Torsion angles (°)		
Sn–C(1)–C(2)–C(3)	102.4	95.5

[a] e.s.d.s in parentheses.

(4) Description of the Structures

The most relevant structural parameters of the ϕ_3Sn(trimethylallyl) complexes **3** and **4** are listed in Table III. The atom numbering schemes are shown in Fig. 1. Both structures are very similar to that reported for the unmethylated analogue [12], all showing nearly undistorted tetrahedral coordination about the tin atoms, with the phenyl groups taking on a paddle wheel conformation. The C–C single bonds in **3** and **4** are of typical length (1.508(5) and 1.510(6) Å, respectively), in contrast to the abnormally short C–C single bond (1.38(3) Å) in unmethylated ϕ_3Sn(allyl) [12]. The C=C double bond in **3** is of typical length (1.320(5) Å), but it is significantly shortened in **4** (1.268(7) Å) such that it is close to the length reported for unmethylated ϕ_3Sn(allyl) (1.24(3) Å) [12]. The torsion angles about CHR–C(Me)= (102.4° for **3**, 95.5° for **4**) are also close to that reported for unmethylated ϕ_3Sn(allyl) (108° and 97° for the two independent molecules in the structure) [12]. All of these torsion angles are reasonably close to the 90° predicted by theoretical calculation to lead to the maximum amount of σ–π conjugation between the Sn–C and C=C bonds [19]. Any dπ–pπ bonding between the metal and the allyl double bond is ruled out by the long distance from the midpoint of the double bond to the tin (~3.40 Å for **3** and **4**), as it is for unmethylated ϕ_3Sn(allyl) [12].

Supplementary Material

Tables IV and V listing structure amplitudes (×10), $10|F_o|$ *versus* $10|F_c|$ for compounds **3** and **4**, respectively, have been deposited with the Editor-in-Chief.

Acknowledgements

Acknowledgment is made to the Donors of The Petroleum Research Fund, administered by the American Chemical Society, for the partial support of this research. This work was also supported, in part, through a Cottrell College Science Grant of Research Corporation.

References

1 G. Erker, K. Berg, C. Kruger, G. Muller, K. Angermund, R. Benn and G. Scroth, *Angew. Chem., 23,* 455 (1984).
2 M. A. Hendrik, P. J. Lemaire and F. Jellinek, *J. Organomet. Chem., 14,* 149 (1968).
3 W. J. Highcock, R. M. Mills, L. J. Spencer and P. Woodward, *J. Chem. Soc., Dalton Trans.,* 829 (1986).
4 J. Nieman, J. W. Pattiasina and J. H. Teuben, *J. Organomet. Chem., 262,* 157 (1984).
5 H. J. de Liefde Meijer and F. Jellinek, *Inorg. Chim. Acta, 4,* 651 (1970).
6 A. Zwijnenburg, H. O. Van Oven, C. J. Groenenboom and H. J. de Liefde Meijer, *J. Organomet. Chem., 94,* 23 (1975).
7 J. Blenkers, H. J. de Liefde Meijer and J. H. Teuben, *J. Organomet. Chem., 14,* 149 (1968).
8 D. Seyferth and M. A. Weiner, *J. Org. Chem., 26,* 4797 (1961).
9 J. A. Ibers and W. C. Hamilton (eds.), 'International Tables for X-Ray Crystallography', Vol. 4, International Union of Crystallography, Kynoch Press, Birmingham, U.K., 1974.
10 J. C. Huffman, L. N. Lewis and K. G. Caulton, *Inorg. Chem., 19,* 2755 (1980).
11 W. P. Neumann, 'The Organic Chemistry of Tin', Wiley, New York, 1970, p. 122.
12 D. Furlani, D. Marton, G. Tagliavini and G. Valle, *J. Organomet. Chem., 293,* 207 (1985).
13 G. Tagliavini, V. Peruzzo and G. Plazzogna, *Inorg. Chim. Acta, 24,* L47 (1977).
14 A. Gambaro, G. Peruzzo, G. Plazzogna and G. Tagliavini, *J. Organomet. Chem., 197,* 45 (1980).
15 V. Peruzzo and G. Tagliavini, *J. Organomet. Chem., 162,* 37 (1978).
16 J. A. Mangravite, *Organomet. Chem. Rev., 7,* 45 (1979), and refs. therein.
17 Yu. P. Egorov, L. A. Leites, I. D. Kravtsova and V. F. Mironov, *Akad. Nauk, SSSR, Otdel. Khim. Nauk.,* 1114 (1963).
18 C. M. Van Zyl, J. L. McKeeby, P. C. Van Dort, E. J. Larson, M. E. Silver and J. C. Huffman, unpublished results.
19 R. D. Bach and P. A. Scherr, *Tetrahedron Lett., 13,* 1099 (1973).