PREPARATION AND REACTIONS OF 1,2-BIS-(TRIMETHYLTIN)TETRAPHENYLBENZENE AND RELATED COMPOUNDS. A NOVEL BIMOLECULAR ELIMINATION OF TRIMETHYLTIN IODIDE

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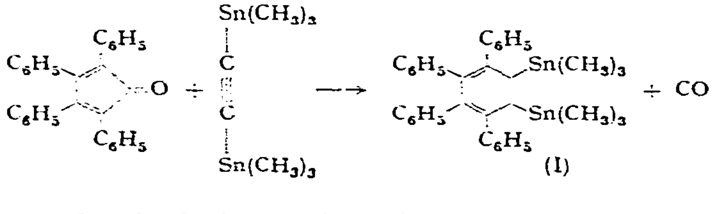
Our interest in unsaturated organometallic compounds[1] has extended to compounds in which metal atoms are linked directly to acetylenic substituents. In this paper we report on the Diels-Alder reaction of bis(trimethyltin)acetylene with tetraphenylcyclopentadienone (tetracyclone) and on some reactions of the resulting 1,2-bis-(trimethyltin)tetraphenylbenzene and its derivatives.

The reaction of bis(organomercuri)acetylenes with substituted cyclopentadienones has been reported previously by Abramov and Shapshinskaya[2], *e.g.*, the reaction of tetracyclone with bis(methylmercuri)acetylene, which gave 1,2-bis(methylmercuri)tetraphenylbenzene with concomitant loss of carbon monoxide. However, reactions of such 1,2-bis(organomercuri)aromatics were not studied, nor have such cyclopentadienone–alkynylmetal reactions been extended to other metal acetylene systems by these or other workers.

A reaction of tetracyclone with bis(trimethyltin)acetylene (1:1 mole ratio) in refluxing xylene for 138 h resulted in evolution of carbon monoxide and gave 1,2-bis(trimethyltin)tetraphenylbenzene (I) in virtually quantitative yield. Similar reactions of tetracyclone with trimethyl(phenylethynyl)tin and with trimethylpropynyltin resulted in formation of (trimethyltin)pentaphenylbenzene (II) and 1-(trimethyltin)-2-methyltetraphenylbenzene (III) respectively.



(I)

The cleavage of aryl substituents from tin by halogens occurs readily and at a rate considerably faster than that of alkyl–tin cleavage by halogens[3]. Thus, barring complicating steric factors, (I), (II) and (III) should serve as useful starting materials

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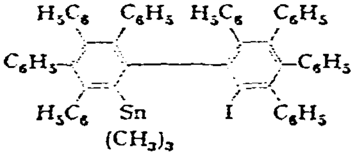
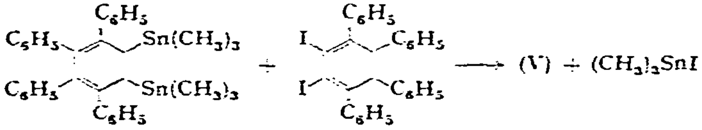
for the preparation of the corresponding dihalo- and monohalobenzenes. This was found to be the case. Treatment of (I) with chlorine, bromine and iodine gave the respective 1,2-dihalotetraphenylbenzenes in quantitative yield. In a similar fashion, brominolysis of (II) resulted in bromopentaphenylbenzene, while cleavage of (III) with HBr gave 1-methyl-2,3,4,5-tetraphenylbenzene.

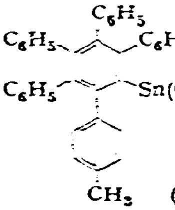
Of particular interest to us was 1-iodo-2-(trimethyltin)tetraphenylbenzene (IV) as a potential source of tetraphenylbenzyne via trimethyltin iodide elimination. It has been shown that photolysis of 1-iodo-2-(iodomercuri)benzene or of bis-(*o*-iodophenyl)mercury produces benzyne (trapping experiments with tetracyclone)[4], and it seemed possible that (IV) could react in a similar manner. A different reaction, however, was observed when (IV) [prepared by reaction of (I) with one equivalent of iodine] was heated at 220–230° with an equimolar amount of tetracyclone. Trimethyltin iodide (*ca.* 49 % based on the desired reaction) indeed was evolved and isolated. The other product, however, was not octaphenylnaphthalene, rather it was 2-iodo-2'-(trimethyltin)octaphenylbiphenyl (V) (81 % yield). The same reaction was

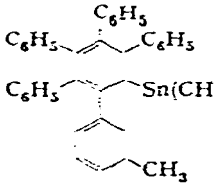

(V)

observed when (IV) was heated at 230° by itself. The formation of (V) possibly could result from a bimolecular condensation of (IV) with itself with elimination of trimethyltin iodide. That this most probably was the case was shown by the preparation of (V) in 53 % yield by the following reaction:



Further proof that we were dealing with a bimolecular coupling reaction was given by the preparation of (trimethyltin)pentaphenylbenzene in 52 % yield by the reaction of (I) with iodobenzene. The product of this reaction was identical with the product from the tetracyclone–(phenylethynyl)trimethyltin reaction.


(VI)


(VII)

The reactions of (I) with *p*-iodotoluene and with *m*-iodotoluene gave in good yield two different 1-(trimethyltin)-2-tolyltetraphenylbenzenes, (VI) and (VII) respectively, indicating that this reaction was specific in terms of the position of aryl coupling. (VI) is poorly soluble in ether and chloroform; (VII) is very soluble in these solvents. A mixture melting point of (VI) (m.p. 289°) and (VII) (m.p. 284–286°) was depressed (254–257°). The infrared spectra of (VI) and (VII) were similar, but not superimposable.

This reaction then represents a novel biphenyl synthesis and a new reaction in organotin chemistry. Similar reactions are known to occur with more electropositive metal derivatives, *e.g.*, the reaction of *o*-bromophenyllithium with *o*-dibromobenzene in tetrahydrofuran solution, which gives 2,2′-dibromobiphenyl in 74% yield^5, but are new in the organic chemistry of heavy metals. (The aryltin and aryllithium reactions need not be, and indeed, probably are not, related to each other mechanistically.) However, the aryl iodide–aryltin coupling reaction appears to have only limited applicability when less hindered organotin systems are involved, at least under these reaction conditions. Thus, for instance, the reaction of (*o*-fluorophenyl)trimethyltin with iodobenzene at reflux temperature gave 2-fluorobiphenyl in only 12% yield, and the reaction of trimethylphenyltin with iodobenzene produced trimethyltin iodide in only 11% yield after 24 h at 180–190°. The attempted preparation of *p*-terphenyl by the reaction of (*p*-biphenylyl)trimethyltin with iodobenzene was not successful. Under the conditions of the experiment (excess iodobenzene as solvent, 19 h at reflux) some disproportionation appeared to occur: Bis(biphenylyl)dimethyltin was isolated in 22% yield and tetramethyltin was detected using gas chromatography. Finally, all attempts to eliminate trimethyltin iodide from (V), thermally or photolytically, to give octaphenylbiphenylene, were unsuccessful.

The apparently limited applicability of the aryltrimethyltin–aryl iodide coupling reaction has prompted us to terminate our efforts in this area for the time being. The observed reaction, 2(IV) → (V) + $(CH_3)_3SnI$, however, may be of some importance in connection with the question of benzyne generation from *o*-haloaryl-metal compounds, and studies in this direction are continuing.

EXPERIMENTAL

General comments

All reactions involving organotin-substituted acetylenes were carried out under an atmosphere of prepurified nitrogen. Melting points are uncorrected. Analyses were performed by Dr. S. M. NAGY, M.I.T. Microchemical Laboratory, and by the Galbraith Laboratories, Inc., Knoxville, Tenn. NMR spectra were determined using a Varian Associates A60 NMR spectrometer; chemical shifts are given in p.p.m. downfield from tetramethylsilane.

Bis(trimethyltin)acetylene

Sodium acetylide was prepared in liquid ammonia from 0.63 g-atom of sodium^6. To the reagent was added 0.49 mole of trimethyltin chloride in 70 ml of ether. The mixture was stirred for 30 min; then the Dry Ice condenser was removed and the ammonia allowed to evaporate slowly. The solid residue was treated with 700 ml of ether and the mixture was filtered. The filtrate was evaporated at —70° (1 mm) and the residue was distilled at 90–100° (0.2 mm) to give 70 g of bis(trimethyltin)acetylene (81%), m.p. 59–60°. (Found: C, 27.10; H, 5.16. $C_8H_{18}Sn_2$ calcd.: C, 27.32; H, 5.16%.)

1. *Diels-Alder reactions*

1,2-Bis(trimethyltin)tetraphenylbenzene (I). A solution of 25 g (0.071 mole) of bis(trimethyltin)acetylene and 26.5 g (0.069 mole) of tetracyclone in 100 ml of xylene was heated at reflux for 138 h. The solvent was distilled away *in vacuo* to leave 48.4 g (99 %) of solid residue. Several recrystallizations from acetone gave 44 g (90 %) of (I), m.p. 232–233°. (Found: C, 60.87; H, 5.51; Sn, 33.67; mol. wt., 701. $C_{36}H_{38}Sn_2$ calcd.: C, 61.07; H, 5.41; Sn, 33.52 %; mol. wt., 708.)

A similar reaction [0.052 mole of tetracyclone, 0.071 mole of bis(trimethyltin)-acetylene] carried out in 100 ml of toluene for 138 h at reflux gave (I) in 86 % (recrystallized) yield. A reaction carried out in a sealed tube at 160–180° for 67 h (0.02 mole of each reactant, no solvent) gave pure (I) in 50 % yield.

(Trimethyltin)pentaphenylbenzene (II). A solution of 15.7 mmoles of tetracyclone and 16.1 mmoles of trimethyl(phenylethynyl)tin in 100 ml of xylene was heated at reflux with stirring for 126 h. Removal of the solvent at reduced pressure left a black-red residue. Recrystallization from methylene chloride–acetone gave 8.0 g (82 %) of white solid, m.p. 320–326°. Two further recrystallizations gave white microcrystals, m.p. 325–328°. (Found: C, 75.17; H, 5.63. $C_{39}H_{34}Sn$ calcd.: C, 75.38; H, 5.52 %.)

1-Trimethyltin-2-methyltetraphenylbenzene (III). A solution of 14.6 mmoles of tetracyclone and 17.7 mmoles of propynyltrimethyltin in 100 ml of xylene was heated at reflux for 147 h. The solvent was removed *in vacuo* and the residue chromatographed (Woelm alumina, neutral, activity 1; 4:1 hexane–benzene) to give 5.56 g (68 %) of white solid, m.p. 179–186°. Two recrystallizations from methylene chloride–methanol gave pure (III), m.p. 193–196°. (Found: C, 73.00; H, 5.74. $C_{34}H_{32}Sn$ calcd.: C, 73.01; H, 5.77 %.) NMR ($CDCl_3$): singlets at —0.05, 2.30, 6.77 and 7.10 p.p.m., relative area ratio 9:3:10:10 respectively.

2. *Cleavage reactions*

1,2-Dichlorotetraphenylbenzene. Five mmoles (3.45 g) of (I) was added to a solution of 0.8 g of chlorine in 90 g of carbon tetrachloride. The resulting solution was stirred at 0° for 1 h. Volatiles ware removed *in vacuo* to leave 2.25 g (100 %) of 1,2-dichlorotetraphenylbenzene, m.p. 243–244°. Two recrystallizations from chloroform–acetone gave pure material, m.p. 246–247° (lit.[7] m.p. 242°). (Found: C, 79.70; H, 4.82; Cl, 15.55. $C_{30}H_{20}Cl_2$ calcd.: C, 79.79; H, 4.46; Cl, 15.75 %.)

1,2-Dibromotetraphenylbenzene. A solution of 15 mmoles of bromine in 10 ml of carbon tetrachloride was added to a solution of 7.5 mmoles of (I) in 17 ml of CCl_4 at —20°. The bromine was consumed rapidly. After the mixture had been stirred for 30 min at room temperature, the volatiles were removed *in vacuo*. The residue, 4.15 g (100 %), m.p. 282–284°, was recrystallized from chloroform–acetone to give pure product, m.p. 284–285°. (Found: C, 66.44; H, 3.75; Br, 29.70. $C_{30}H_{20}Br_2$ calcd.: C, 66.68; H, 3.73; Br, 29.59 %.)

1,2-Diiodotetraphenylbenzene. Three mmoles of (I) in 20 ml of chloroform was treated at room temperature for 20 h with 6 mmoles of iodine in 90 ml of chloroform. Removal of volatiles left the product in quantitative yield; m.p. 308–309° (yellow colored and sintering above 302°) after recrystallization from methylene chloride. (Found: C, 56.68; H, 3.30; I, 39.93. $C_{30}H_{20}I_2$ calcd.: C, 56.80; H, 3.18; I, 40.02 %.)

1-Iodo-2-(trimethyltin)tetraphenylbenzene (IV). To 6 mmoles of (I) in 30 ml of chloroform at 0° was added with stirring during 6 h a solution of 6 mmoles of iodine in 50 ml of chloroform. Removal of volatiles left (IV) in quantitative yield; m.p. 218° (from ether–acetone) with slow decomposition. (Found: C, 59.18; H, 4.44; I, 18. 77; mol. wt., 657. $C_{33}H_{29}ISn$ calcd.: C, 59.05; H, 4.36; I, 18.90%; mol. wt., 672.)

Bromopentaphenylbenzene. A solution of 1.96 mmoles of (II) in 100 ml of methylene chloride was treated with 2 mmoles of bromine in 20 ml of carbon tetrachloride. The resulting yellow solution was stirred at room temperature for 72 h until the color was discharged. Evaporation of volatiles left 0.98 g (90%) of crude product, m.p. 274–277°. Several recrystallizations from methylene chlorine–methanol gave m.p. 289–291°. (Found: C, 79.88; H, 4.69. $C_{36}H_{25}Br$ calcd.: C, 80.44; H, 4.78%.)

1-Methyl-2,3,4,5-tetraphenylbenzene. Anhydrous hydrogen bromide (22 g) was dissolved in 200 ml of methanol and 2.46 mmoles of (III) in 50 ml of ether was added. The mixture was stirred for 20 h at room temperature. Removal of volatiles left 0.93 g of white crystals, m.p. 170–174°. Two recrystallizations from ether–pentane gave material of m.p. 177–178°. (Found: C, 93.67; H, 6.29. $C_{31}H_{24}$ calcd.: C, 93.90; H, 6.10%.) NMR ($CDCl_3$ solution): 2.14 p.p.m. (singlet); 6.75 p.p.m. (doublet); 7.04 p.p.m. (doublet); 7.33 p.p.m. (singlet) with relative areas 3:10:10:1, in agreement with the expected structure. The literature[8] m.p., however, is 185–186°.

3. *Thermal condensations*

Attempted reaction 1-iodo-2-(trimethyltin)tetraphenylbenzene with tetracyclone. A 50 ml flask containing 3.6 mmoles of (IV) and 3.6 mmoles of tetracyclone was connected to a trap cooled to —78°. The flask contents were heated under nitrogen to 220–230° for 2 h, then the system (flask and trap) was evacuated and sealed off. After 4 h at 220–230°, the system was brought back to atmospheric pressure. The trap contained 0.5 g of liquid which was identified as trimethyltin iodide. The residue in the flask (3.27 g) was chromatographed (Woelm alumina, neutral, activity 1; 1:1 benzene–hexane). A white, crystalline solid, (V), m.p. 273° 1.53 g (81%) was thus obtained. (Found: C, 71.87; H, 4.77; I, 12.30; mol. wt., 1031. $C_{63}H_{49}ISn$ calcd.: C, 71.95; H, 4.70; I, 12.07%; mol. wt., 1051.6.) UV (heptane): λ_{max} 247 mμ ($\varepsilon = 4805$).

UV irradiation (Hanovia Lamp Division Cat. No. 30600) of 10 mmoles of tetracyclone and 5 mmoles of (IV) in 200 ml of benzene at reflux in a quartz flask for 8 h resulted in no definite products. Sixty % of (IV) was recovered.

Thermal decomposition of 1-iodo-2-(trimethyltin)tetraphenylbenzene. (IV), 8.3 mmoles (5.6 g), was decomposed at 230–240° in the system used above using the same general procedure. The trimethyltin iodide trapped at —78° (1.0 g) was equivalent to an 84% yield. The solid residue was chromatographed as above to give 0.4 g (7.2%) of (IV) and 3.0 g of (V) (69%), m.p. 273°.

In another experiment 2 mmoles of (IV) was decomposed at 230° in the same manner. The condensed trimethyltin iodide was treated with 10% aqueous-alcoholic KF solution. The precipitated trimethyltin fluoride was filtered, washed, dried and weighed. The yield was 0.15 g (91%).

Reaction of 1,2-diiodotetraphenylbenzene with 1,2-bis(trimethyltin)tetraphenylbenzene. In the system described above was placed 2.06 mmoles each of the reactants. The flask was maintained at 230–245°, one hour at atmospheric pressure, then 1 h *in vacuo*. Trimethyltin iodide was formed in 77.4% yield. The residue was chromatographed as above to give 260 mg (16.8 %) of (I), 1.1 g (52.5 %) of (V) and a mixture of (V) and 1,2-diiodotetraphenylbenzene. A mixed m.p. of (V) from this reaction and (V) from the reaction above was not depressed.

Reaction of iodobenzene with 1,2-bis(trimethyltin)tetraphenylbenzene. In the usual reaction system was heated at reflux a solution of 5 mmoles of (I) in 8.14 g of iodobenzene. Removal of excess iodobenzene and the trimethyltin iodide *in vacuo* left 3.28 g of dry residue. The latter was fractionally recrystallized from methylene chloride–ether to give 1.6 g (51.5 %) of (II), m.p. 321–324°, and 1.00 g (28.2 %) of recovered (I). A mixture m.p. of (II) from this reaction and (II) from the Diels-Alder preparation was not depressed.

Reaction of p-iodotoluene with 1,2-bis(trimethyltin)tetraphenylbenzene. A solution of 5 mmoles of (I) in 7.6 g of *p*-iodotoluene was heated at reflux for 5 h. The excess *p*-iodotoluene and the trimethyltin iodide were removed *in vacuo*. The residue was washed carefully with ether. The solid which remained was recrystallized from methylene chloride–ether to give 2.2 g (70 %) of (VI), m.p. 289°. (Found: C, 75.70; H, 5.81. $C_{40}H_{36}Sn$ calcd.: C, 75.61; H, 5.71 %.)

Reaction of m-iodotoluene with 1,2-bis(trimethyltin)tetraphenylbenzene. The same procedure as that used for *p*-iodotoluene was followed in the reaction of 5 mmoles of (I) with 8.0 g of *m*-iodotoluene. The residue after removal of volatiles was chromatographed (Woelm alumina, neutral, activity 1; 1:5 ether–pentane) to give 0.6 g (17 %) of (I) and 2.05 g (63 %) of (VII), m.p. 284–286° (from ether–methanol). (Found: C, 75.86; H, 5.78. $C_{40}H_{36}Sn$ calcd.: C, 75.61; H, 5.71 %.)

Reaction of iodobenzene with (o-fluorophenyl)trimethyltin. A mixture of 10 mmoles of (*o*-fluorophenyl)trimethyltin and 20 mmoles of iodobenzene was heated at reflux for 22 h. Volatile material was removed *in vacuo* by a bulb-to-bulb distillation. From the residue was isolated by recrystallization from ethanol–water 210 mg (12 %) of 2-fluorobiphenyl, m.p. 74° (lit.[9] m.p. 73.5°). (Found: C, 83.20; H, 5.25; F, 11.68. $C_{12}H_9F$ calcd.: C, 83.69; H, 5.27; F, 11.04 %.)

Reaction of iodobenzene with trimethylphenyltin. A mixture of 30 mmoles of trimethylphenyltin and 32 mmoles of iodobenzene was heated at reflux (185–190°) for 24 h. The reaction mixture was diluted with 120 ml of diethyl ether and treated for 1 h with dry, gaseous ammonia. A precipitate, $(CH_3)_3SnI \cdot NH_3$ or $(CH_3)_3SnI \cdot 2NH_3$ (ref. 11), 1.13 g, resulted.

Reaction of iodobenzene with (4-biphenylyl)trimethyltin. A solution of 10 mmoles of (4-biphenylyl)trimethyltin in 9 g of iodobenzene was heated at reflux for 19 h. Volatiles were removed by bulb-to-bulb distillation *in vacuo*. The residue was recrystallized from methylene chloride–ether. White crystals, 0.6 g, m.p. 173–174°, were isolated. Analysis indicated that they were bis(4-biphenylyl)dimethyltin. (Found: C, 68.52; H, 5.54. $C_{26}H_{24}Sn$ calcd.: C, 68.61; H, 5.32 %.) Tetramethyltin was shown by gas chromatography to be present in the distillate.

Attempted elimination of trimethyltin iodide from (V). In the usual system was placed 2.1 g (2 mmoles) of (V) and heated under nitrogen at 290–300° for 30 min. The substance melted, became yellow, then dark brown. Further heating at 220° *in vacuo* followed. The residue after this treatment weighed 1.9 g, and 0.18 g had condensed in the −78° trap. The condensate contained trimethyltin iodide. Attempted purification of the dark brown residue by chromatography was not successful, no crystalline products being obtained. Several similar attempts failed.

4. *New aryltrimethyltin compounds*

(o-Fluorophenyl)trimethyltin was prepared by the reaction of *o*-fluorophenyllithium[10] and trimethyltin chloride. To 92 ml of 1.1 *N* *n*-butyllithium in pentane at —78° under nitrogen was added over *ca.* 8 min 17.5 g (0.1 mole) of *o*-bromofluorobenzene (Aldrich Chemical Co.) in 170 ml of ether. The temperature was maintained below —50°. After the addition was completed a solution of 20 g (0.1 mole) of trimethyltin chloride in 200 ml of ether at —75° was added during 5 min. The temperature was not allowed to exceed —45°. The reaction mixture was stirred at —78° for 4 h, then was allowed to warm to room temperature overnight. It was hydrolyzed with saturated ammonium chloride solution. The ether layer was dried, treated with dry, gaseous ammonia and fractionally distilled to give 15.6 g (60 %) of (*o*-fluorophenyl)trimethyltin, b.p. 65° (4 mm), n_D^{25} 1.5180. (Found: C, 41.53; H, 5.04. $C_9H_{13}FSn$ calcd.: C, 41.75; H, 5.06 %.)

(4-Biphenylyl)trimethyltin. To a solution of 17.6 g (0.075 mole) of 4-bromobiphenyl in 140 ml of ether was added 72 ml of 1.05 *N* *n*-butyllithium in pentane. The resulting mixture was stirred at room temperature for 2.5 h and then 0.075 mole of trimethyltin bromide in 25 ml of ether was added. The reaction mixture was heated at reflux for 1 h and stirred at room temperature overnight. A work-up procedure similar to that described above gave 11.9 g (50 %) of (4-biphenylyl)trimethyltin, b.p. 118–119° (0.25 mm), m.p. 46–47°. (Found: C, 56.34; H, 5.45. $C_{15}H_{18}Sn$ calcd.: C, 56.83; H, 5.85 %.)

ADDENDUM

Two Diels-Alder reactions between tetracyclone and ethynylsilanes are reported here, although the products were not investigated further.

1-(Trimethylsilyl)-2,3,4,5-tetraphenylbenzene

A solution of 16.8 mmoles of ethynyltrimethylsilane* and 10 mmoles of tetracyclone in 14 g of toluene was heated at reflux for 165 h. Volatiles were distilled away *in vacuo*, leaving 4.4 g (96.6 %) of residue. Recrystallization from ether–ethanol gave 3.8 g (90 %), m.p. 200°. (Found: C, 87.19; H, 6.2. $C_{33}H_{30}Si$ calcd.: C, 87.17; H, 6.65 %.)

1-(Dimethylphenylsilyl)-2,3,4,5-tetraphenylbenzene

A solution of 17.1 mmoles of ethynyldimethylphenylsilane* and 1.6 mmoles of tetracyclone in 14.7 g of xylene was heated at reflux for 43 h. Removal of volatiles *in vacuo* left 5.4 g of product, which after recrystallization from ether-ethanol had m.p. 175°. (Found: C, 88.02; H, 6.21. $C_{38}H_{32}Si$ calcd.: C, 87.75; H, 6.55 %.)

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SUMMARY

The Diels-Alder reaction between tetracyclone and the appropriate tin- and silicon-

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* Prepared in these Laboratories by Dr. D. E. WELCH.

substituted acetylene has produced the following compounds: 1,2-bis(trimethyltin)-tetraphenylbenzene; (trimethyltin)pentaphenylbenzene; 1-(trimethyltin)-2-methyl-tetraphenylbenzene; 1-(trimethylsilyl)-2,3,4,5-tetraphenylbenzene; 1-(dimethyl-phenylsilyl)-2,3,4,5-tetraphenylbenzene. Cleavage reactions of the tin compounds with halogens are described. A novel coupling reaction between an aryltrimethyltin compound and an aryl iodide has been discovered. As an example is cited the reaction of 1,2-bis(trimethyltin)tetraphenylbenzene with 1,2-diiodotetraphenylbenzene at 240°, which gives 2-iodo-2'-(trimethyltin)octaphenylbiphenyl.

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