the ionic additions, *exo*-2-bromo-*endo*-2-chloronorbornene (1), was separated by preparative vpc.

Anal. Calcd for $C_7H_{10}BrCl$: C, 40.13; H, 4.81. Found: C, 40.27; H, 4.77.

2,3-Dibromonorbornane (8).—To a homogeneous solution of 56.59 g of norbornene and 195.9 g of 1,2-dibromotetrachloroethane in 500 ml of warm carbon tetrachloride was added 1.00 g of azoisobutyronitrile. After 5 min of irradiation with a G.E. sun lamp, vigorous exothermic reaction began, and the light was turned off. After reaction had subsided, the light was turned on, and the solution was refluxed on the steam bath for 8 hr. The nmr spectrum of an aliquot at this time showed no vinylic absorption. After carbon tetrachloride and tetrachloroethylene were removed at the rotary evaporator, 2,3-dibromonorbornane (8) distilled as a colorless oil: yield 145 g (95%); bp 83–85° (3.5 mm) [lit.[14] bp 55° (0.4 mm)]; the nmr spectrum (CCl_4) indicated this to be a mixture of the 2,3-*trans*- and 2,3-*exo,cis*-dibromides, in the ratio of 97:3.[11]

exo-**2-Chloro-***endo***-2-bromonorbornane (2).**—A solution of 2.17 g of 2-bromonorbornene (7), prepared from **8**,[18] in 20 ml of methylene chloride was hydrochlorinated at −78° for 3 hr by a standard procedure.[17] The product was washed with water and dilute aqueous sodium bicarbonate, and the solvent was removed at the rotary evaporator to afford 2.66 g of a pale yellow oil. Nmr analysis showed this to contain 2.40 g (92%) of **2**, contaminated with a small amount of methylene chloride. Distillation afforded 2.08 g (80%) of the chlorobromide **2**: bp 42–49° (0.06 mm); mp 34.5–36.0°. Analysis by vpc (2 ft, 10% Zonyl E-7 on Fluoropak 80, 100°) indicated a 98:2 mixture of **2** and a substance of similar retention time. Dihalide **2** was purified by preparative vpc.

Anal. Calcd for $C_7H_{10}BrCl$: C, 40.13; H, 4.81. Found: C, 40.42; H, 4.81.

Dehydrohalogenation of 1.—To a solution of 50 mg of **1** in 0.1 ml of dry dimethyl sulfoxide in an nmr sample tube was added a small amount (*ca.* 8 mg) of potassium *t*-butoxide. The mixture immediately became yellow. Analysis by nmr showed vinyl absorption at τ 4.15 (d, J = 3.2 cps). The vinyl proton in **3** (CCl_4) appears as a doublet at τ 4.33 (J = 3.2 cps); that of **7** is a doublet at τ 3.83 (J = 3.1 cps). Analysis by vpc under conditions (SE-30, 20% on 60–80 mesh Chromosorb W, 5 ft, 130°) where **3** and **7** are cleanly separated indicated the presence of only **3** and a small amount of unreacted **1**.

Dehydrohalogenation of 2.—Dehydrohalogenation of **2** by the preceding procedure and analysis by vpc indicated the presence of **3** (13%) and **7** (87%).

3-*exo*-**Deuterio-2,2-dichloronorbornane.**—Although the addition valve of the analytical unit[17] could be used, we have substituted the valve of the preparative apparatus, connecting the valve to a glass-tipped syringe containing deuteriosulfuric acid *via* a flexible Teflon needle piercing the rubber septum of the valve. Ten grams of sodium chloride (dried at 110°) was added to the lower, 125-ml flask, and 5 ml of deuteriosulfuric acid was placed in the syringe. The 50-ml reaction flask was chilled to −78° and flushed with deuterium chloride using 1 ml of deuteriosulfuric acid, and 3.2 g of 2-chloronorbornene in 1 ml of methylene chloride was injected. Addition began immediately. After uptake of deuteriosulfuric acid had ceased, the system was returned to room temperature and the dichloride was purified by preparative gas chromatography. Mass spectral analysis indicated an isotopic purity of 98.1%.

Registry No.—**1,** 21690-94-8; **2,** 21690-95-9.

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Alkyltin Bond Cleavage–Cyclization by 1,4-Dilithio-1,2,3,4-tetraphenylbutadiene[1a]

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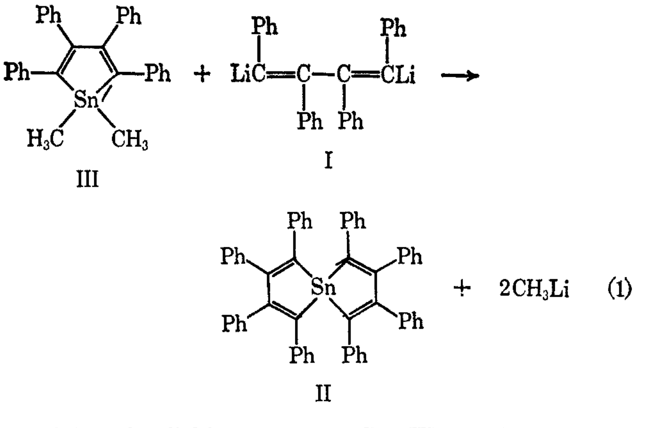
1,4-Dilithio-1,2,3,4-tetraphenylbutadiene (I) has been employed in an apparently general reaction with organometallic and organometalloidal di- and tetrahalides to yield monocyclic and spiro heterocyclopentadienes (metalloles), respectively.[2–4]

We find that the addition of dimethyl- or diethyltin dichloride to ether suspensions of I results in the formation of octaphenyl-1,1′-spirobistannole (II), whereas divinyltin dichloride gives the expected monocyclic products. Divinyltin dichloride undergoes cleavage by I in THF; diphenyltin dichloride resists cleavage in either solvent. The lability series is thus ethyl and methyl >vinyl >phenyl. 1,1-Dimethyl-2,3,4,5-tetraphenylstannole (III) added to the organodilithium reagent also reacts to give the spiro compound, as do trimethyl- and tri-*n*-butyltin chlorides.

When exactly 1 molar equiv of dimethyltin dichloride was added to I, 1,4-bis(trimethylstannyl)-1,2,3,4-tetraphenylbutadiene (IV) and trimethyltin chloride were identified as minor products along with II. No tetramethyltin was found.

Two routes to the spirobistannole are available, alkyltin bond cleavage–cyclization by 1,4-dilithio-1,2,3,4-tetraphenylbutadiene to produce methyllithium (eq 1), or the disproportionation of III, perhaps catalyzed by the lithium reagent I. We prefer the first route in view of the following observations: (a) the apparent absence of tetramethyltin in the product, (b) isolation of trimethyltin chloride, (c) detection of IV, the likely product of the action of trimethyltin chloride on the dilithio reagent, (d) isolation of the spiro compound in greater than 50% yield, (e) cleavage of tri-



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[1] (a) Presented at the 154th National Meeting of the American Chemical Society, Chicago, Ill., Sept 1967, Abstract No. 024, and at the Octavo Congreso Peruano de Quimica, Cuzco, Peru, Oct 1968; (b) to whom all inquiries should be directed.

[2] F. C. Leavitt, T. A. Manuel, and F. Johnson, *J. Amer. Chem. Soc.*, **81**, 3163 (1959); F. C. Leavitt, T. A. Manuel, F. Johnson, L. U. Matternas, and D. S. Lehman, *ibid.*, **82**, 5099 (1960).

[3] H. Gilman, S. G. Cottis, and W. H. Atwell, *ibid.*, **86**, 1596 (1964).

[4] E. H. Braye, W. Hübel, and I. Caplier, *ibid.*, **83**, 4406 (1961).

alkyltin chlorides to give the spiro compound, and (f) the detection of acetic acid after hydrolysis of a reaction mixture which had been carbonated.

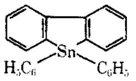
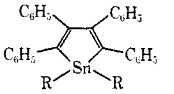
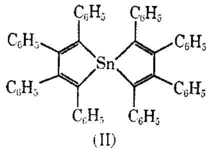
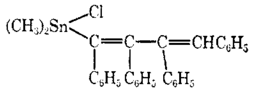
It should be noted that in the reactions where the organodilithium reagent is added to the halide, the latter is always in excess and the expected heterocyclopentadienes are formed.[2-4] In the reverse addition employed here the organodilithium reagent is always present in excess. Diphenyllead dichloride gives the expected monocyclic compound only, as do dimethyldichlorosilane and -germane, no matter what the sequence of addition.

The fission of carbon to tin bonds by organolithium reagents (nucleophilic attack at tin)[5] has been known for some time;[5-8] the cleavage of carbon–tin bonds by hydrogen halides, carboxylic acids, halogens, etc. (electrophilic attack at carbon with possible nucleophilic assistance in some cases) establishes a series of preference with the ease of cleavage phenyl >vinyl >methyl >ethyl.[9]

^{119m}Sn Mössbauer spectroscopy proved useful as a routine adjunct to these studies. One synthesis of III[2] gave as major product a white solid (mp, 196–197°; lit. values for the anticipated stannole 193–195°[10] and 192–193°[2]) whose ir spectrum correlated well with those of the analogous silicon and germanium compounds and whose nmr spectrum contained the expected methyltin and phenyl proton resonances in the approximately correct integrated peak ratio (3:10). The ^{119m}Sn Mössbauer spectrum displayed on an oscilloscope within an hour of sample mounting, however, showed a well-defined quadrupole splitting (see Table I). This splitting rules out unambiguously the anticipated stannol structure,[11] and the compound was identified as (1,2,3,4-tetraphenyl-*cis,cis*-1,3-butadienyl)dimethyltin chloride.[10]

TABLE I

^{119m}SN MÖSSBAUER PARAMETERS, MM/SEC[a]

Compound	Isomer shift	Quadrupole splitting
	1.20 ± 0.06	
 R = CH_3(III)	1.23 ± 0.06[b]	
R = $CHCH_2$	1.25 ± 0.06	
R = C_6H_5	1.19 ± 0.06	
 (II)	1.30 ± 0.06	
 $(CH_3)_2Sn(Cl)C(C_6H_5)=C(C_6H_5)-C(C_6H_5)=CHC_6H_5$	1.27 ± 0.06[c]	2.68 ± 0.12[c]
$(CH_3)_2SnCl_2$	1.53[d]	3.41[d]
	1.68[e]	3.85[e]
$(CH_2{=}CH)_2SnCl_2$	1.46	3.08
$(C_6H_5)_2SnCl_2$	1.31[d]	2.66[d]
	1.52[e]	2.98[e]
	1.4[f]	2.8[f]

[a] With respect to an $^{119m}SnO_2$ source; all compounds studied at liquid nitrogen temperature. [b] An average of six determinations of different reaction products and donated samples. [c] An average of four determinations of intermediates from different reactions. [d] R. H. Herber, H. A. Stöckler, and W. T. Reichle, *J. Chem. Phys.*, **42**, 2447 (1965). The authors assign an error of ±0.03 mm/sec. [e] M. Cordey-Hayes, *J. Inorg. Nucl. Chem.*, **26**, 2306 (1964). The values have been converted from those for the gray tin source used. The author assigns an error of ±0.05 mm/sec. [f] V. A. Bryukhanov, V. I. Goldanskii, N. N. Delyagin, L. A. Korytko, E. F. Makarov, I. P. Suzdalev, and V. S. Shpinel, *Soviet Phys. JETP*, **16**, 321 (1963). The authors assign an error of ±0.3 mm/sec. See also, A. Yu. Aleksandrov, Ya. G. Dorfman, O. L. Lependina, K. P. Mitrofanov, M. V. Poltnikova, L. S. Polak, A. Ya. Tempkin, and V. S. Shpinel, *Russ. J. Phys. Chem.*, **38**, 1185 (1964). These authors assign an error of ±0.05 mm/sec.

Experimental Section

Analytical Procedures.—Our constant acceleration cam-drive Mössbauer spectrometer has been previously described.[12] Data refinement was carried out at the LaJolla, Calif. Computer Center and the Cornell Computer Center. Nmr spectra were recorded on a Varian A-60 spectrometer; infrared spectra on Perkin-Elmer Infracords 137 and 337. All operations were carried out under nitrogen and at room temperature. Microanalytical determinations were performed by Galbraith Laboratories, Inc. Molecular weight determinations were by vapor phase osmometry in benzene. Some low resolution mass spectra were obtained on a Consolidated Model 21-103A; high resolution spectra were obtained on an A.E.I. MS-9 instrument.

Cleavage Reaction with Dimethyltin Dichloride.—Dimethyltin dichloride (0.13 mol) in 100 ml of anhydrous THF was added dropwise to I (0.7 mol) prepared by the method of Gilman, *et al.*,[3] in 120 ml of ether. After the addition was *ca.* two-thirds completed, the color of the solution turned from dark green to yellow-brown. Upon addition of water (250 ml) a yellow compound (10 g) precipitated. Sublimation at 225° *in vacuo* gave II, mp 263° in 67% yield (based on I which is obtained with an average yield of 65%),[2,3] and a gray residue (1 g) which contained tin, but no aromatic groups, and is most likely a hydrolysis product of dimethyltin dichloride.

Anal. Calcd for $C_{56}H_{40}Sn$: C, 80.78; H, 4.81; Sn, 14.28; mol wt, 831. Found: C, 80.78; H, 4.89; Sn, 14.31; mol wt, 810.

In a second reaction, volatiles were stripped after the addition of dimethyltin dichloride and fractional distillation and chromatography yielded a compound, identified as trimethyltin chloride by mass spectrometry and the superposition of glpc peaks. Sublimation *in vacuo* of the solid residue at 160–165° gave diphenylacetylene and a green solid from which some III was separated by recrystallization from methylene chloride–cyclohexane. A polyisotopic molecular ion corresponding to the mass distributions of a ditin species was found at highest mass (centered at 684) in the mass spectrum of the solid, apparently the parent ion of 1,4-bis(trimethylstannyl)-1,2,3,4-tetraphenylbutadiene (IV). A ditin fragment at P − 15 was of sufficient intensity to be studied by high resolution techniques (obsd: 669.0790; calcd for $C_{33}H_{35}{}^{118}Sn{}^{120}Sn$: 669.0776). Tin containing fragments corresponding to the loss of trimethyltin and an additional methyl group were observed at 521 and 506. Subsequent reactions run in ether alone also resulted in cleavage.

1,1-Dimethyl-2,3,4,5-tetraphenylstannole (III).—Compound I was added slowly to dimethyltin dichloride (0.059 mol) in 250 ml of ether to give III, mp 191.5–192.5°. This material

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(1 g) was dissolved in hot methylene chloride–petroleum ether (bp 60–70°). Upon cooling the solution and careful evaporation of solvent, four fractions of crystals were obtained, mp 192–193, 195–204, 195–206, 204–206°, respectively. The Mössbauer spectrum of each sample was a singlet resonance at 1.23 ± 0.06 mm/sec. The highest melting fraction was a pale yellow-green solid. This experiment was repeated with an authentic sample of III, prepared as in ref 2 with identical results.

Anal. Calcd for $C_{30}H_{26}Sn$: C, 71.35; H, 5.19; Sn, 23.50; mol wt, 505. Found: C, 71.86; H, 5.21; Sn, 23.12, Cl, 0.00; mol wt, 516.

A similar reaction produced a white solid (mp, 196–197°) in 70% yield whose nmr spectrum contained a singlet resonance at τ 9.44 and the phenyl multiplet centered at 3.0. The ^{119m}Sn Mössbauer spectrum was a doublet. The (1,2,3,4-tetraphenyl-*cis,cis*-1,3-butadienyl)dimethyltin chloride[10] structure is consistent with these data.

Anal. Calcd for $C_{30}H_{27}SnCl$: C, 66.51; H, 5.02; Cl, 6.55; Sn, 21.9; mol wt, 541.2. Found: C, 65.94; H, 4.92; Cl, 5.13; Sn, 21.95; mol wt, 521.

Octaphenyl-1,1′-spirobistannole (II) from 1,1-Dimethyl-2,3,4,5-tetraphenylstannole (III).—Compound III (0.008 mol) in 60 ml of anhydrous THF was slowly added to 60 ml of a suspension of I (0.023 mol) and stirred for 3 hr under nitrogen. After addition of water the organic layer gave a yellow solid (1 g, 25% yield based on I) identical with II. None of the stannole compound could be isolated from the remaining oil.

Cleavage Reaction of Diethyltin Dichloride.—Diethyltin dichloride (0.032 mol) in 150 ml of THF was added to I (0.075 mol) in 200 ml ether. Near the end of the addition the solution turned from a dark green to a deep yellow-brown. After 1 hr of stirring, carbon dioxide was passed through the solution, producing a short-lived, deep red color. Water was added and the organic layer was separated and dried over calcium sulfate. The solvent was removed to give II after recrystallization from methylene chloride–petroleum ether (4 g, 13% yield based on I). The remaining oil resisted further recrystallization.

1,1-Divinyl-2,3,4,5-tetraphenylstannole.—To I (0.075 mol) in 200 ml of ether was slowly added divinyltin dichloride (0.041 mol) in 100 ml of ether. After 1 hr of stirring, the solvent was removed and 15 g of yellow solid (42% yield based on I) was isolated. The residue was recrystallized from methylene chloride and washed with several portions of 1:1 benzene–petroleum ether to give pale yellow crystals, mp 154–156°. None of the more insoluble spiro compound was found. The mass spectrum contained a series of peaks corresponding in distribution of intensities to the relative abundances of the tin isotopes with the *m/e* value of the ^{120}Sn peak = 530; calcd for $C_{32}H_{26}Sn$: 530. The ir spectrum contained prominent bands at 3000, 1580, 1475, 1000, 950, 790, 780, 760, 700, and 478 cm^{-1}. Subsequent reactions run in THF gave a yellow solid, mp 265–270°, with ir and nmr spectra identical with that of an authentic sample of II.

Anal. Calcd for $C_{32}H_{26}Sn$: C, 72.59; H, 4.91; Sn, 22.49; mol wt, 529. Found: C, 73.29; H, 5.10; Sn, 21.57; Cl, 0.00; mol wt, 570.

Cleavage Reaction of Trimethyltin Chloride.—Compound I (0.075 mol) was prepared as described above, excess lithium chips were removed, and 450 ml of THF was added. Trimethyltin chloride (0.057 mol) was added in 200 ml of ether. A yellow solid formed near the end of the addition whose ir and nmr spectra were identical with those of II (25 g, 80% yield based on I, mp 268–270°). Solvents were vacuum evaporated and some tetramethyltin was isolated by fractional distillation.

Cleavage Reaction of Tri-*n*-butyltin Chloride.—Compound I (0.075 mol) was prepared as described above, excess lithium chips were removed, and 450 ml of THF was added. Tri-*n*-butyltin chloride (0.074 mol) dissolved in 200 ml of ether was added dropwise, but the green color of the dilithium reagent remained until additional tri-*n*-butyltin chloride (0.077 mol) was added, at which time a yellow color appeared. Water (50 ml) was added and the organic layer separated to yield II (mp 265–270°, 8 g, 27% yield based on I).

Hexaphenylplumbole.—An ethereal suspension of diphenyllead dichloride (300 ml, 0.02 mol) was mixed with I (200 ml, 0.05 mol) in ether and stirring was continued for 4 days. Water (100 ml) was added, and yellow crystals (1.83 g, 15% yield based on diphenyllead dichloride), mp 153–155°, were obtained after evaporation of the ether layer and recrystallization from methylene chloride. Sublimation *in vacuo* and column chromatography on silica gel resulted in decomposition. The ir spectrum contained prominent bands at 3090, 1610, 1575, 1490, 1443, 1075, 1020, 1000, 781, 763, 738, and 687 cm^{-1} and was very similar to that of the tin analog.

Anal. Calcd for $C_{40}H_{30}Pb$: C, 66.95; H, 4.18; mol wt, 717. Found: C, 66.6; H, 4.16; mol wt, 770.

1,1-Dimethyl-2,3,4,5-tetraphenylgermole.—This white crystalline compound, mp 183–184°, prepared by the method of Gilman, *et al.*,[3] was recrystallized from petroleum ether (bp 60–70°) and sublimed *in vacuo* at 175°. The nmr spectrum contained a resonance at τ 9.37 in addition to that owing to the phenyl protons, and the ir spectrum showed prominent bands at 3100, 3040, 1605, 1490, 1445, 1080, 1035, 834, 819, 789, 709, and 697 cm^{-1}. High resolution mass spectral data gave for the monogermanium molecular ion at mass 458 an exact mass of 460.1239 based on ^{72}Ge (calcd for $C_{30}H_{26}{}^{72}Ge$: 460.1244).

Anal. Calcd for $C_{30}H_{26}Ge$: C, 78.50; H, 5.66; Ge, 15.83. Found: C, 78.32; H, 5.80; Ge, 15.91.

Registry No.—I, 21289-08-7; II, 21779-48-6; III, 20195-60-2; dimethyltin dichloride, 753-73-1; diethyltin dichloride, 866-55-7; divinyltin dichloride, 7532-85-6; 1,1-divinyl-2,3,4,5-tetraphenylstannole, 21779-52-2; trimethyltin chloride, 1066-45-1; tri-*n*-butyltin chloride, 1461-22-9; hexaphenylplumbole, 21779-54-4; 1,1-dimethyl-2,3,4,5-tetraphenylgermole, 20991-88-2; (1,2,3,4-tetraphenyl-*cis,cis*-1,3-butadienyl)dimethyltin chloride, 21779-56-6; 9,9-diphenyl-9-stannafluorene, 5381-63-5; hexaphenylstannole, 21813-34-3.

Acknowledgments.—N. W. G. Debye and S. E. Ulrich recorded the ^{119m}Sn Mössbauer spectra. We are grateful to Dr. E. H. Braye of European Research Associates, S. A., Brussels, Belgium, and Dr. F. C. Leavitt of the Eastern Research Laboratories of the Dow Chemical Co., Framingham, Mass., for the donation of several samples of the derivatives and to Dr. R. Weiss of the Monsanto Company, St. Louis, Mo., for a sample of 9-stannafluorene. This work was supported by the Advanced Research Projects Agency.

A Synthesis of Norisotuboflavine

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The β-carboline alkaloids flavocarpine (**1**),[1] tuboflavine (**2**),[2] isotuboflavine (**3**),[3] norisotuboflavine (**4**),[3] and methyl β-carboline-1-carboxylate (**5**)[3] are minor constituents of *Pleiocarpa tubicina* Benth. Syntheses of flavocarpine,[1] tuboflavine,[4] and methyl β-carboline-1-carboxylate[5] have been reported. In this note we describe a synthesis of norisotuboflavine (**4**) from the companion alkaloid **5**. Very recently Rosenkranz and Schmid reported a synthesis of **4** from 4,5-dihydrocanthin-6-one,[6] which is available synthetically in low yield from DL-tryptophan.[4,6] Inasmuch as carboline

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