2,3-benzo-5-butyl-1,1,4,4-tetraethyl-1,4-disilacyclohexa-2,4-diene (7) and 4 were produced in 67% and 5% yields, respectively. Pure 7 was isolated by preparative GLC: MS *m/e* 301 (M^+ – Et); IR 1560, 1414, 1377, 1231, 1127, 1001 cm^{-1}; ^1H NMR 0.67–0.89 (m, 20 H, EtSi), 0.91–0.96 (t, 3 H, CH_3, J = 7.1 Hz), 1.29–1.55 (m, 4 H, CH_2CH_2), 2.15–2.26 (m, 2 H, CH_2), 6.61 (br s, 1 H, HC=C), 7.29–7.52 ppm (m, 4 H, phenylene ring protons); ^{13}C NMR 5.95, 6.35, 7.64, 7.69 (EtSi), 14.1, 22.7, 30.6, 38.9 (C_4H_9), 127.6, 127.8, 133.1, 133.3, 143.2, 143.7 (phenylene ring carbons), 139.5 (HC=C), 162.2 ppm (C=CH): ^{29}Si NMR –15.08, –13.37 ppm. Anal. Calcd for $C_{20}H_{34}Si_2$: C, 72.65; H, 10.36. Found: C, 72.44; H, 10.23.

Thermolysis of 1 with Dimethyl Acetylenedicarboxylate. A mixture of 0.1555 g (0.627 mmol) of 1 and 0.2011 g (1.42 mmol) of dimethyl acetylenedicarboxylate was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 5,6-benzo-2,3-dicarbomethoxy-1,1,4,4-tetraethyl-1,4-disilacyclohexa-2,3-dicarbomethoxy-1,1,4,4-tetraethyl-1,4-disilacyclohexa-2,5-diene (8) (83% yield) and 7% of the starting compound 1. Pure 8 was isolated by preparative GLC: MS *m/e* 390 (M^+); IR 1714, 1462, 1454, 1433, 1217, 1121 cm^{-1}; ^1H NMR 0.81–0.95 (m, 20 H, EtSi), 3.78 (s, 6 H, MeO), 7.39–7.55 ppm (m, 4 H, phenylene ring protons); ^{13}C NMR 5.95, 7.42 (EtSi), 51.9 (MeO), 128.5, 133.3, 140.8 (phenylene ring carbons), 153.9 (C=C), 169.9 ppm (C=O); ^{29}Si NMR –9.55 ppm. Anal. Calcd for $C_{20}H_{30}O_4Si_2$: C, 61.49; H, 7.74. Found: C, 61.46; H, 7.80.

Thermolysis of 1 with Diphenylacetylene. A mixture of 0.1707 g (0.688 mmol) of 1 and 0.2912 g (1.63 mmol) of diphenylacetylene was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 4 (55% yield) and unchanged 1 (20% yield).

Thermolysis of 1 with 3-Octyne. A mixture of 0.1633 g (0.659 mmol) of 1 and 0.2573 g (2.33 mmol) of 3-octyne was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 4 (32% yield) and unchanged 1 (64% yield).

Thermolysis of 1 with 1-Hexene. A mixture of 0.1707 g (0.688 mmol) of 1 and 0.0992 g (1.18 mmol) of 1-hexene was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 4 (68% yield).

Thermolysis of 1 with 2,3-Dimethyl-1,3-butadiene. A mixture of 0.2236 g (0.902 mmol) of 1 and 0.2921 g (3.56 mmol) of 2,3-dimethyl-1,3-butadiene was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 4 (38% yield) and unchanged 1 (43% yield).

Thermolysis of 1 with Benzaldehyde. A mixture of 0.2841 g (1.15 mmol) of 1 and 0.9136 g (8.62 mmol) of benzaldehyde was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 5,6-benzo-3-phenyl-1,1,4,4-tetraethyl-2-oxa-1,4-disilacyclohexa-2,5-diene (9) (52% yield) and 4 (12% yield). Pure 9 was isolated by preparative GLC: MS *m/e* 354 (M^+); IR 1456, 1120, 1026, 830 cm^{-1}; ^1H NMR 0.98–1.09 (m, 20 H, EtSi), 5.18 (s, 1 H, HC), 7.30–7.55 ppm (m, 9 H, phenyl and phenylene ring protons); ^{13}C NMR 1.82, 2.59, 6.45, 6.72, 6.81, 7.11, 7.28, 7.51 (EtSi), 67.9 (HC), 124.4, 125.4, 128.1 (2 C), 128.2, 132.8, 134.0, 141.8, 143.4, 143.7 ppm (phenyl and phenylene ring carbons); ^{29}Si NMR –8.51, 7.10 ppm. Anal. Calcd For $C_{21}H_{30}OSi_2$: C, 71.12; H, 8.53. Found: C, 70.95; H, 8.45.

Thermolysis of 1 with Paraformaldehyde. A mixture of 0.1813 g (0.731 mmol) of 1 and 0.1325 g (4.41 mmol for HCHO) of paraformaldehyde was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 5,6-benzo-1,1,4,4-tetraethyl-2-oxa-1,4-disilacyclohexa-2,5-diene (10) (88% yield). Pure 10 was isolated by preparative GLC: MS *m/e* 278 (M^+); IR 1461, 1226, 1108, 1002, 973 cm^{-1}; ^1H NMR 0.74–0.99 (m, 20 H, EtSi), 3.91 (s, 2 H, H_2C), 7.33–7.51 ppm (m, 4 H, phenylene ring protons); ^{13}C NMR 3.88, 6.27, 6.90, 7.48 (EtSi), 53.9 (H_2C), 128.2 (2 C), 132.7, 133.7, 142.5, 143.4 ppm (phenylene ring carbons); ^{29}Si NMR –11.10, 6.98 ppm. Anal. Calcd for $C_{15}H_{26}OSi_2$: C, 64.68; H, 9.41. Found: C, 64.62; H, 9.41.

Thermolysis of 1 with Acetone. A mixture of 0.1124 g (0.453 mmol) of 1 and 0.0513 g (0.884 mmol) of acetone was heated at 250 °C in a sealed tube for 24 h. The reaction mixture was analyzed by GLC as being 4 (30% yield) and unchanged 1 (45% yield).

Thermolysis of 1 with Methyl Vinyl Ketone. A mixture of 0.2406 g (0.970 mmol) of 1 and 0.1342 g (1.92 mmol) of methyl vinyl ketone was heated in a sealed tube at 250 °C for 24 h. The reaction mixture was analyzed by GLC as being 7,8-benzo-1,1,6,6-tetraethyl-3-methyl-2-oxa-1,6-disilacycloocta-3,7-diene (11) (30% yield) and 4 (57% yield). Pure 11 was isolated by preparative GLC: MS *m/e* 318 (M^+); IR 1418, 1318, 1168, 1013 cm^{-1}; ^1H NMR 0.77–1.00 (m, 20 H, EtSi), 1.54 (br d, 2 H, H_2C, J = 7.9 Hz), 1.75 (br s, 3 H, Me), 4.71 (br t, 1 H, HC=C, J = 7.9 Hz), 7.29–7.62 ppm (m, 4 H, phenylene ring protons); ^{13}C NMR 6.20, 6.86, 7.10, 7.73 (EtSi), 10.5 (H_2C), 22.0 (Me), 103.5 (HC=C), 127.5, 128.2, 134.5, 135.5, 142.4, 144.4 (phenylene ring carbons), 146.7 ppm (C=CH). Anal. Calcd for $C_{18}H_{30}OSi_2$: C, 67.86; H, 9.49. Found: C, 67.69; H, 9.49.

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A Triptycene Complex of Tin(II): $[(C_{20}H_{14})SnCl(AlCl_4)]_2$

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Triptycene ($C_{20}H_{14}$) reacts with equimolar quantities of anhydrous $SnCl_2$ and $AlCl_3$ in benzene to give a crystalline complex $[(C_{20}H_{14})SnCl(AlCl_4)]_2\cdot 2C_6H_6$. [Triclinic, space group $P\bar{1}$, a = 10.404 (3) Å, b = 13.343 (3) Å, c = 11.194 (2) Å, α = 98.63 (2)°, β = 109.23 (2)°, γ = 105.77 (2)°, V = 1375.3 Å^3, Z = 1, R_W = 0.032 for 298 refined parameters and 3597 unique reflections with $F_o \geq 4.0\sigma(F_o)$.] According to the X-ray crystal structure analysis, the compound features discrete centrosymmetrical dinuclear complex units, with each tin atom η^6-coordinated to two neighboring benzene rings of a triptycene ligand but at different metal–ring distances (2.90 and 3.38 Å). The angle between the two coordinated arene rings is contracted to 108.2°, as compared to 120° in the parent hydrocarbon. The ψ-octahedral coordination sphere of the Sn(II) centers is completed by two tin-bridging chlorine atoms and a chelating tetrachloroaluminate ligand. The benzene of crystallization is not engaged in metal complexation. A lead(II) complex $[(C_{20}H_{14})Pb\cdot(AlCl_4)_2\cdot 0.5C_6H_6]$ has also been prepared and characterized by analytical data.

Introduction

Triptycene is a very special hydrocarbon with regard to its ligand properties for metals: In a relatively rigid molecular geometry of point group D_{3h} symmetry, it features three eqivalent benzene rings mutually inclined by 120° angles.[1] At first glance, it would appear that this molecule

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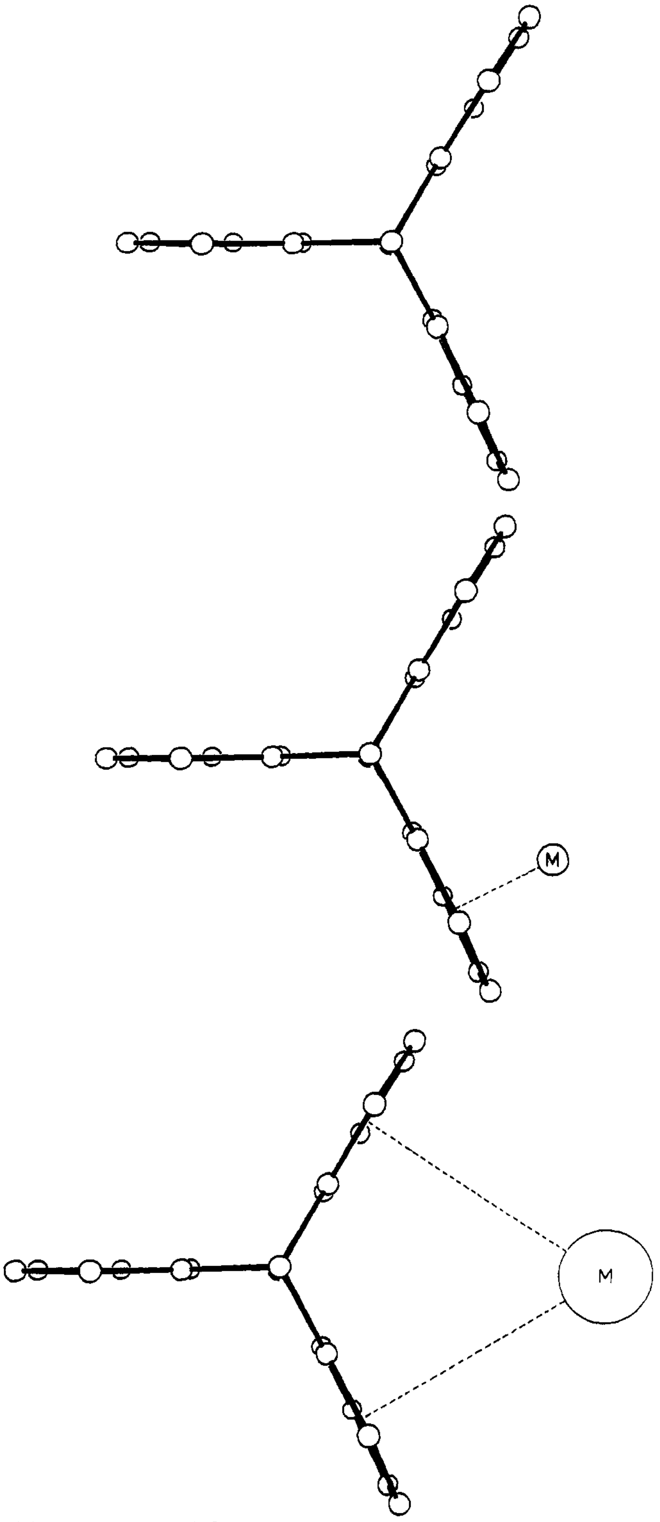

Figure 1. (a, top) Structure of triptycene, projected along the 3-fold axis of the molecule. (The plot was drawn with published data.[1]) (b, middle) η^6-Metal coordination to one ring of triptycene, as in $(C_{20}H_{14})Cr(CO)_3$. (The plot was generated with published structural data for this complex.[2]) (c, bottom) Positioning of a metal atom in double η^6-coordination to triptycene. (The crossing point of the two normals to the planes in the centers of the rings was selected as the metal site. As a result, very long ring-to-metal distances are obtained).

can offer opportunities for a wide variety of donor–acceptor interactions (Figure 1). A closer investigation shows, however, that the details are not really very favorable: While metal coordination to only one side of one of the *o*-xylylene rings meets with sterical hindrance originating from the requirements of the remainder of the ligand sphere of the metal (Figure 1b), the bent sandwich coordination to two adjacent rings suffers from relatively long metal–ring distances owing to the large inter-arene angle of 120° (Figure 1c). It thus becomes obvious that bent sandwich complexation according to the arrangement in Figure 1c would first of all require a very large metal atom.

Only very few π-complexes of triptycene have been reported in the literature,[2-5] and only for two of these has the molecular structure been determined. The tricarbonylchromium(0) adduct is a monoarene complex (Figure 1b), in which the complexation leads to only a small widening of the inter-arene angle to 122.1°, as sterical crowding in this region of the molecule is not too great with the relatively small, rodlike carbonyl ligands.[2] From a previous study of the $Cr(CO)_3$ complex of 9,10-dihydroanthracene, it even appears that there might be some van der Waals attraction between carbonyl and arene ligands.[6] The tetranuclear cluster unit $Co_4(CO)_9$ accommodates a triptycene ligand in much the same type of arrangement but with a wider inter-arene angle (124.1°) owing to the bulkiness of the cobalt carbonyl cluster fragment.[2] Other products, as obtained from triptycene and $(NH_3)_3Co(CO)_3$ or $Hg(O_2CCF_3)_2$, have not yet been fully characterized.[4,5]

Monoarene complexation of main group metals is well-documented from investigations in the 1970s.[7] However, it was only in the course of our more recent studies[8-12] that the first examples of bis- and tris(arene) complexes of some of these metals including the bent sandwich type could also be detected.[13-16] From species

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with a nonforced ligand geometry, the optimum metal–arene distances thus became known quite accurately for the cations Ga(I), In(I), Tl(I), and Sn(II). While a whole series of compounds of this type are available[17-19] for the heavier triad of low-valent group 13 metals, only a very few representatives have been obtained with Ge(II)[12] and Sn(II),[11] and none have been obtained with Pb(II).

Based on the above considerations, it is now felt that tryptycene might offer just the right ligand geometry for bis(arene) coordination of the two quite large dications Sn(II) and Pb(II). It is important to note in this context that for these elements centered η^6-bonding to arenes (hexahapto) is the only mode of arene coordination observed to date. In order to enhance the acceptor properties of the metals, counterions with poor donor properties, like the tetrachloroaluminate anions, have to be introduced, following a concept successfully employed already very early on in the π-complex chemistry of main group elements.[7]

Preparation and Properties of the Adducts

For the preparation of the compound, benzene solutions of triptycene and anhydrous tin(II) or lead(II) dichloride in a molar ratio of 1:1 (or greater) are heated to reflux temperature, and the vapors are passed through the reservoir of a Soxhlet apparatus filled with an excess of anhydrous aluminum(III) trichloride. From the orange solutions obtained in this procedure, red crystalline products are precipitated after concentration under vacuum and on slow cooling. When the material is redissolved in benzene, red (Sn) and green (Pb) solutions are formed, respectively, but colorless crystals can finally be isolated in good yield (Sn, 64%; Pb, 66%).

$$2SnCl_2 + (AlCl_3)_2 + 2C_{20}H_{14} \rightarrow [(C_{20}H_{14})SnCl(AlCl_4)]_2$$

$$PbCl_2 + (AlCl_3)_2 + C_{20}H_{14} \rightarrow [(C_{20}H_{14})Pb(AlCl_4)_2]$$

Satisfactory elemental analyses and ^1H NMR data confirm the stoichiometry of the adducts and the presence of unchanged triptycene moieties, respectively. Due to the solubility characteristics of the compounds, the NMR spectra could only be obtained for solutions in strongly polar media like acetone or dimethyl sulfoxide, for which extensive dissociation of the complexes has to be assumed. It is therefore of no surprise that the proton resonances are similar to those of the free triptycene in its maximum molecular symmetry (D_{3h}). The infrared spectra of the complexes are also quite similar and indicate analogous structures (Experimental Section).

The two compounds are sensitive to moisture and undergo rapid hydrolysis upon exposure to the laboratory atmosphere. The tin compound melts at 183 °C with decomposition, but no well-defined decomposition temperature could be observed for the lead analogue. Crystalline material after heating in vacuo or in a stream of dry nitrogen is free of crystal benzene, but single crystals obtained from benzene solutions and carefully protected against loss of solvate molecules contain 1 mol of crystal benzene for the monomeric stoichiometric unit. The crystal structure of such single crystals of the tin compound has been determined by X-ray diffraction.

Table I. Fractional Atomic Coordinates and Equivalent Isotropic Thermal Parameters for the Title Compound[a]

atom	x/a	y/b	z/c	U(eq), Å^2
Sn	0.05612 (4)	0.40112 (3)	0.39392 (3)	0.044
Al	0.3085 (1)	0.3095 (1)	0.6396 (1)	0.039
Cl1	−0.1786 (1)	0.41583 (9)	0.4348 (1)	0.046
Cl2	0.3615 (1)	0.3878 (1)	0.5006 (1)	0.059
Cl3	0.0915 (1)	0.3119 (1)	0.6086 (1)	0.059
Cl4	0.4546 (2)	0.3987 (1)	0.8307 (1)	0.052
Cl5	0.2965 (1)	0.1485 (1)	0.5967 (1)	0.066
C1	−0.2067 94)	0.2692 (3)	0.0298 (4)	0.037
C2	0.0332 (4)	0.2320 (3)	0.0602 (4)	0.032
C10	−0.0737 94)	0.3706 (3)	0.0895 (4)	0.036
C11	0.0551 (4)	0.3511 (3)	0.1055 (4)	0.032
C12	0.1874 (4)	0.4335 (3)	0.1686 (4)	0.038
C13	0.1901 (5)	0.5367 (3)	0.2162 (4)	0.034
C14	0.0619 (5)	0.5562 (3)	0.1998 (4)	0.044
C15	−0.0717 (5)	0.4729 (3)	0.1368 (4)	0.042
C20	−0.1743 (4)	0.2004 (3)	0.1254 (4)	0.030
C21	−0.0453 (4)	0.1806 (3)	0.1419 (4)	0.030
C22	0.0049 (5)	0.1244 (3)	0.2302 (4)	0.039
C23	−0.0763 (5)	0.0879 (4)	0.3017 (4)	0.049
C24	−0.2032 (5)	0.1080 (4)	0.2868 (4)	0.048
C25	−0.2529 (5)	0.1652 (3)	0.1990 (4)	0.039
C30	−0.2049 (5)	0.2128 (3)	−0.0981 (4)	0.032
C31	−0.0753 (5)	0.1938 (3)	−0.0817 (4)	0.033
C32	−0.0547 (5)	0.1450 (3)	−0.1890 (4)	0.039
C33	−0.1647 (6)	0.1160 (4)	−0.3118 (4)	0.047
C34	−0.2926 (6)	0.1344 (4)	−0.3288 (4)	0.044
C35	−0.3138 (5)	0.1828 (4)	−0.2210 (4)	0.043
C40	0.5607 (5)	−0.2858 (4)	−0.1044 (5)	0.048
C41	0.5444 (5)	−0.2859 (4)	−0.2306 (5)	0.044
C42	0.5874 (6)	−0.1920 (5)	−0.2642 (5)	0.072
C43	0.6502 (6)	−0.0953 (4)	−0.1670 (6)	0.058
C44	0.6670 (5)	−0.0959 (4)	−0.0400 (5)	0.050
C45	0.6216 (5)	−0.1909 (4)	−0.0096 (5)	0.055

[a] $U(\mathrm{eq}) = [U(1)U(2)U(3)]^{1/3}$, where $U(1)$, $U(2)$, and $U(3)$ are the eigenvalues of the $\mathbf{U}(ij)$ matrix. Esd's are in parentheses.

Table II. Selected Bond Distances (Å) and Angles (deg) for the Title Compound[a]

Distances			
Sn–Cl1	2.680 (1)	Sn–Cl3	2.807 (1)
Sn–Cl1′	2.578 (1)	Sn–Z1	2.90
Sn–Cl2	3.070 (1)	Sn–Z2	3.38
Angles			
Cl1–Sn–Cl1′	82.1 (1)	Sn–Cl1–Sn′	97.9 (1)
Interplane Angles			
C10–C15/C20–C25	108.18	Sn–Z1/C10–C15	3.42[b]
C20–C25/C30–C35	125.81	Sn–Z2/C20–C25	13.20[c]

[a] For atomic numbering, see Figure 2; Z, ring center. [b] Angle between line connecting the tin atom and the center Z1 of the ring C10–C15 and the normal to the least-squares plane of this ring. [c] As under footnote *a* but referring to Z2 and the ring C20–C25.

Molecular Structure of Chloro(triptycene)tin(II) Tetrachloroaluminate Dimer. The structure of the dimeric molecular unit of the crystals is shown in Figure 2 with the atomic numbering scheme employed. This species has a crystallographic center of inversion. The two tin atoms are bridged by two chlorine atoms. The resulting four-membered ring, planar by symmetry, is a strongly distorted square with angles of 82.1 (1)° at tin and of 97.9 (1)° at chlorine and Sn–Cl1/Cl1′ distances of 2.680 (1) and 2.578 (1) Å, respectively. Each tin atom is also chelated by a tetrachloroaluminate counterion with inequivalent distances Sn–Cl2/Cl3 at 3.070 (1) and 2.807 (1) Å.

The ligand sphere of the tin atoms is complete by bis(arene) coordination of a triptycene molecule. The metal appears in a position with strongly inequivalent distances

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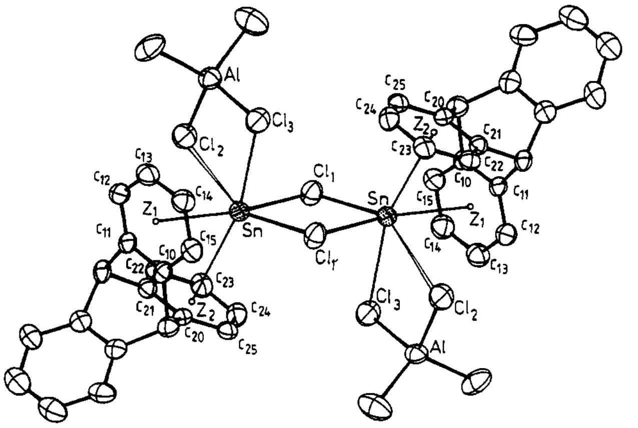

Figure 2. Molecular structure of bis[chloro(triptycene)tin(II) tetrachloroaluminate] with atomic numbering. The dimer has a crystallographic center of inversion. (ORTEP, 50% probability ellipsoids, hydrogen atoms omitted for clarity; the two molecules of benzene of crystallization have also been excluded.)

from the two neighboring rings (2.90 and 3.38 Å, respectively). Nevertheless, the bonding to each ring can be described as hexahapto (η^6), as indicated by the small angles between the normal of the ring plane and the line connecting the metal and the center of the ring (Sn–Z1 3.42°, Sn–Z2 13.2°). Another important structural criterion is the *reduced* inter-arene angle of the benzene rings engaged in tin coordination (108.2°) as compared to the 120° angle in free triptycene.[1] The remaining inter-arene angles are widened accordingly to 125.8 and 126.0° (Table II). This finding is a clear indication of bis(arene) coordination. Note that angle *widening* occurs in the monoarene coordination to triptycene with transition-metal complex fragments!

The environment of the tin atoms can thus be described as distorted octahedral, with no direct evidence for stereochemical activity of the lone pair of electrons present at the metal center. This situation is similar to the structural characteristics of the only other bis(arene) complex of tin(II) known, with two independent benzene rings: $[(C_6H_6)_2SnCl(AlCl_4)]_2$. The two compounds differ mainly by the inter-arene angle (108.2° versus 101.9 and 99.7° in the benzene complex) and by a variation in the tetrachloroaluminate connectivity, while the Sn_2Cl_2 core is the same in both cases. The four-membered ring SnCl1Sn′Cl1′ shows Sn–Cl1 and Sn–Cl1′ distances of 2.680 (1) and 2.578 (1) Å (average 2.629 Å), whereas in the bis(benzene) reference complex the average of four such distances is 2.623 Å. Each $AlCl_4$ unit is chelating a single tin atom in the triptycene complex but bridging two tin atoms in the bis(benzene) complex. The benzene of crystallization in the lattice of the title compound has no metal contacts. All intermolecular distances are beyond the van der Waals limits.

Experimental Section

All experiments were carried out in an atmosphere of dry, purified nitrogen. Solvents and glassware were dried and also kept under nitrogen. NMR spectra were recorded on Jeol PMX60 and GX270 spectrometers. IR spectra were recorded on Perkin-Elmer 577 and Nicolet FT-IR-5 DX spectrometers. Melting points (Büchi 510) are uncorrected.

Bis[chloro(triptycene)tin(II) tetrachloroaluminate]. In a Soxhlet apparatus, triptycene (0.90 g, 3.54 mmol) and anhydrous tin(II) dichloride (0.98 g, 5.17 mmol) are dissolved in 175 mL of dry benzene and heated to reflux temperature. The condensed benzene vapors are passed over anhydrous aluminum(III) trichloride (1.33 g, 9.97 mmol) placed in the reservoir of the Soxhlet apparatus. After 3 h, an orange solution is obtained, which is concentrated to 120 mL under vacuum and briefly heated to 60 °C. On cooling, deep red crystals are formed, which are redissolved in 80 mL of dry benzene at reflux temperature. Colorless crystals of $[(C_{20}H_{14})SnCl(AlCl_4)]_2{\cdot}2C_6H_6$ form on cooling (2.96 g, 64% yield), which decompose at 183 °C. Anal. Calcd for $C_{52}H_{40}Al_2Cl_{10}Sn_2$ (1310.76): C, 47.65; H, 3.08; Cl 27.05. Found: C, 46.21; H, 2.97; Cl, 27.49. IR (Hostaflon, Nujol, KBr): 3103 (s), 3081 (s), 3048 (s), 1954 (m), 1811 (w), 1169 (w), 1160 (w), 1040 (w), 1028 (w), 978 (w), 857 (vw), 801 (w), 743 (s), 627 (m), 548 (w), 490 (w), 481 (w) [cm^{-1}]. ^1H NMR (acetone-d_6, 30 °C): 7.39 (m, 3 H, C_6H_4), 7.29 (s, 2 H, C_6H_6), 6.92 (m, 3 H, C_6H_4), 5.58 (s, 1 H, CH).

Bis[chloro(triptycene)lead(II) tetrachloroaluminate]. As described for the tin(II) analogue above, triptycene (0.88 g, 3.46 mmol), lead(II) dichloride (0.96 g, 3.45 mmol), and anhydrous aluminum(III) trichloride (1.84 g, 13.80 mmol) were slowly reacted in 175 mL of dry benzene. The original orange solution turns green as the primary crop of crystals is redissolved in benzene to separate colorless crystals of $[(C_{20}H_{14})Pb(AlCl_4)_2]_2{\cdot}0.5C_6H_6$ upon cooling (3.44 g, 66% yield); slow decomposition occurred on heating. Anal. Calcd for $C_{43}H_{31}Al_4Cl_{16}Pb_2$ (1637.26): C, 31.54; H, 1.91; Cl, 34.65. Found: C, 30.67; H, 2.06; Cl, 33.87. IR (Hostaflon, Nujol, KBr): 3100 (m), 3072 (w), 3037 (m), 1239 (w), 1205 (m), 1171 (w), 1150 (vw), 1123 (w), 1102 (vw), 1042 (vw), 973 (m), 900 (m), 801 (w), 741 (m), 677 (m), 600 (m), 548 (vw), 505 (w), 483 (w), 475 (vw) [cm^{-1}]. ^1H NMR (dimethyl-d_6 sulfoxide, 30 °C): 7.36 (m, 3 H, C_6H_4), 7.27 (s, 2 H, C_6H_6), 6.89 (s, 3 H, C_6H_4), 5.56 (s, 1 H, CH).

Crystal Structure Determination. $[(C_{20}H_{14})SnCl(AlCl_4)]_2{\cdot}(C_6H_6)_2$, $M_{rel} = 1310.76$, triclinic, space group $P\bar{1}$, $a = 10.404$ (3) Å, $b = 13.473$ (3) Å, $c = 11.194$ (2) Å, $\alpha = 98.63$ (2)°, $\beta = 109.23$ (2)°, $\gamma = 105.77$ (2)°, $V = 1375.3$ Å^3, $Z = 1$, $\rho_{calc} = 1.582$ g cm^{-3}, μ(Mo Kα) = 14.67 cm^{-1}, $T = -50$ °C. A suitable single crystal of the complex was sealed under argon at dry ice temperature into a glass capillary and examined directly on the diffractometer (Syntex P2$_1$, Mo Kα radiation, $\lambda = 0.71069$ Å, graphite monochromator). Reduced cell calculations did not indicate any higher cell symmetry. The measured intensities of 4566 reflections were corrected for Lorentz and polarization effects and empirically for absorption effects. From 4566 independent structure factors, 3597 with $F_o \geq 4.0\sigma(F_o)$ were taken as "observed" and used for all calculations. The structure was solved by automated Patterson methods (SHELXS-86) and completed by difference Fourier syntheses. All non-hydrogen atoms were refined by using anisotropic thermal displacement parameters; hydrogen atoms were included into the structure factor calculations with fixed atomic contributions ($U_{iso} = 0.05$ Å^2). Final R and R_w values were 0.036 and 0.032, respectively ($w = 1/\sigma^2(F_o)$, SHELX-76 used for 298 refined parameters, residual electron density +0.60/−0.50 e/Å^3). The fractional atomic coordinates and equivalent isotropic displacement parameters are presented in Table I.

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Supplementary Material Available: Tables listing fractional atomic coordinates, anisotropic thermal parameters, H-atom coordinates, and MPLN calculations (11 pages); a table listing observed and calculated structure factor amplitudes (20 pages). Ordering information is given on any current masthead page.