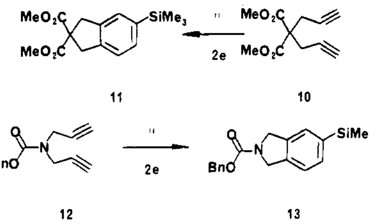
erably more labile in the thermal sense than the phenylethyne derivative and underwent rapid decomplexation to the isomeric tributylbenzenes **5b** and **6b** (**5b**/**6b** = 1.22) at room temperature.[11] That the low-valent niobium center could also function as a catalyst[12a] for alkyne trimerization was demonstrated by the following experiment. Reduction of $CpNbCl_4$ with Mg^0 (1 equiv) in the presence of 1-hexyne (100 equiv) in THF at 25 °C gave the arenes **5b** and **6b** (**5b**/**6b** = 1.22) in 91% isolated yield. A series of terminal alkynes was subsequently subjected to the catalytic low-valent niobium system described above. The results of this study are compiled in Table I.

Cotton has reported that *internal* alkynes are efficiently polymerized in the presence of catalytic quantities of $[(NbCl_3)_2\cdot(\text{tetrahydrothiophene})_3]$.[12b] It is therefore of considerable interest that *representative internal alkynes (e.g., 3-hexyne, 1-phenyl-1-propyne,[13] etc.) were found to undergo neither polymerization nor cyclotrimerization in the presence of the $CpNbCl_4/Mg^0$ catalyst system.* These results strongly suggested that annulation reactions involving the cocyclization of diynes with relatively unhindered internal acetylenes or hindered terminal acetylenes (e.g., Table I, entry **2e**) would be achievable employing the $CpNbCl_4/Mg^0$ system. Accordingly, addition of the diyne **8** over 6 h to a prereduced solution of $CpNbCl_4$ (0.35 equiv[14]) and 3-hexyne (10 equiv) under argon in THF maintained at 50 °C and subsequent stirring for 12 h provided the indan **9** in 80% chromatographed yield. In a similar fashion, the diester **10** and the urethane **12** were cocyclized with (trimethylsilyl)acetylene (**2e**) to furnish the indan **11** and the isoindolene **13** in isolated yields of 74% and 52%, respectively.[15]



The significance of the foregoing results derives from the ability of monoacetylenes less hindered than bis(trimethylsilyl)acetylene to participate selectively in binary annulations involving diynes. By way of contrast, the successful utilization of cobalt-based catalysts (e.g., $CpCo(CO)_2$) is largely restricted to the use of $(Me_3SiC)_2$ as a cocyclization addend.[16,17] The above examples also illustrate the functional group compatibility of this new method for benzocycle annulation.[18]

Additional applications of niobium(III)- and tantalum(III)-alkyne complexes to synthesis will be the topic of future reports from this laboratory.

Acknowledgment. Support for this research by a grant from the National Institutes of Health is gratefully acknowledged.

Supplementary Material Available: Spectral data for the compounds **5a-d**, **6a-d**, **9**, **11**, and **13** (2 pages). Ordering information is given on any current masthead page.

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[11] Evidence has recently been provided for the intermediacy of tantalacyclopentadienes in the cyclotrimerization of alkynes by tantalum(III) complexes: (a) Strickler, J. R.; Wexler, P. A.; Wigley, D. E. *Organometallics* **1988**, *7*, 2067. (b) Bruck, M. A.; Copenhauer, A. S.; Wigley, D. E. *J. Am. Chem. Soc.* **1987**, *109*, 6525.

[12] Early precedence for the trimerization of *terminal alkynes* on low-valent group V centers [e.g., $(NbCl_3)_2(THT)_3$ and $(TaCl_3)_2(THT)_3$] has been provided by Cotton: (a) Cotton, F. A.; Hall, W. T. *J. Am. Chem. Soc.* **1979**, *101*, 5094. (b) Cotton, F. A.; Hall, W. T.; Cann, K. J.; Karol, F. J. *Macromolecules* **1981**, *14*, 233.

[13] Evidence for the generation of the $CpNbCl_2$-(1-phenyl-1-propyne) complex in a stoichiometric run was provided by its hydrolyses with 1 M HCl to furnish (*Z*)-1-phenyl-1-ethene in ~50% yield (GC).

[14] The use of greater than catalytic concentrations of $CpNbCl_4/Mg^0$ is recommended for runs that involve the slow addition of diynes as a result of competitive decomposition of the active Nb species at elevated temperatures. Temperatures of ca. 50 °C are required for internal alkynes or (trimethylsilyl)acetylene to efficiently participate in cocyclizations with representative diynes.

[15] All new compounds have been fully characterized by 300-MHz ^1H NMR and IR spectroscopy and possess satisfactory elemental (C, H) analyses or exact mass.

[16] Vollhardt, K. P. C. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 539. The present methodology is therefore complementary to well-established annulation procedures based on cobalt.

[17] Jonas, K.; Deffense, E.; Habermann, D. *Angew. Chem. Suppl.* **1983**, 1005-1016.

[18] A comprehensive account concerning the utilization of $(Ph_3P)_3RhCl$ for effecting related diyne-monoacetylene cocyclizations has appeared recently. Grigg, R.; Scott, R.; Stevenson, P. *J. Chem. Soc., Perkin Trans. 1* **1988**, 1357.

Arene Complexes of p-Block Elements: $[(\eta^6\text{-}C_6H_6)_2SnCl(AlCl_4)]_2$. The First Bis(arene) Coordination Compound of a Group 14 Element

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Summary: From the reaction of the molten salt $Sn(AlCl_4)_2$ with benzene the dimeric species $[(\eta^6\text{-}C_6H_6)_2SnCl(AlCl_4)]_2\cdot C_6H_6$ (**1**) has been obtained. Compound **1**, which was characterized by X-ray diffraction analysis, represents the first example for double arene complexation of a group 14 post-transition element. Each Sn center is η^6-bonded to two molecules of benzene. The two benzene planes are inclined with respect to each other and form angles of 101.9° (Sn1) and 99.7° (Sn2), respectively. The two $(\eta^6\text{-}C_6H_6)_2Sn$ units of the dimer are linked by two direct chlorine bridges and two 1,3-bidentate $AlCl_4$ groups. The crystal benzene molecule has no metal contacts. The coordination of the tin(II) centers is thus pseudooctahedral, and the entire dimer approaches noncrystallographic C_{2h} symmetry.

It is now well established that most of the low-valent p-block metals are capable of forming coordination compounds with aromatic hydrocarbons. These complexes display a large variety of stoichiometries and structural features, regarding (1) the hapticity of the aromatic rings bound to the metal center, (2) the individual arene:metal ratio in an "open sandwich", "sandwich", "inverse sandwich", or "cage" type arrangement, and (3) the aggregation of these units to yield dimers, tetramers, polymeric chains, or three-dimensional networks. Moreover, the crystalline compounds very often contain different amounts of noncoordinating (interstitial) aromatic hydrocarbons.

Typical examples with neutral arene donors have been described for the complete triads Ga/In/Tl[1] and As/

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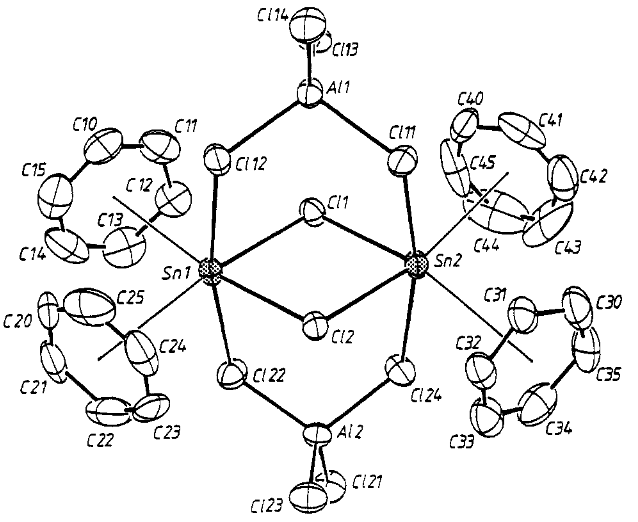

Figure 1. Molecular structure of molecule 1 in the crystal (ORTEP, displacement ellipsoids at 50% probability level). Hydrogen atoms and the interstitial benzene molecule are not shown for clarity.

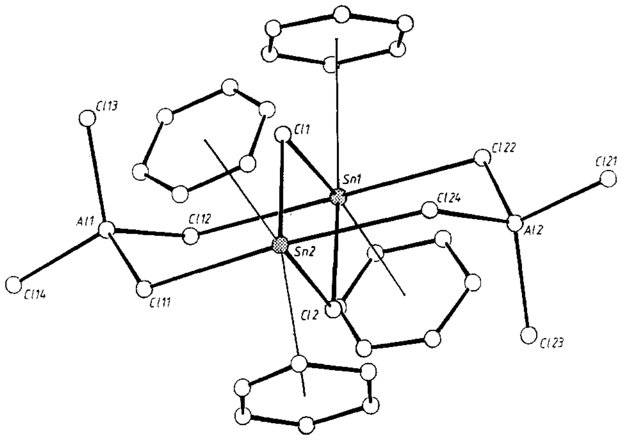

Figure 2. Alternative projection of molecule 1, displaying the eight-membered ring.

Sb/Bi.[2] Depending on the nature of the central atom and the arene component they display most of the structural features mentioned above. By contrast, knowledge on the corresponding compounds of the divalent group 14 elements is restricted to the η^6-benzene and η^6-*p*-xylene monoadducts of $SnCl(AlCl_4)$,[3] which form coordination polymers containing four-membered Sn_2Cl_2 rings, and the η^6-benzene monoadducts of $Sn(AlCl_4)_2$ and $Pb(AlCl_4)_2$,[4] whose structures feature polymeric chains with $AlCl_4$-bridged metal centers. Finally, a tetrameric η^6-hexamethylbenzene adduct of $Sn(AlCl_4)Cl$ could be reported,[5] which contains three molecules of noncoordinating crystal-chlorobenzene. Weak η^6-C_6H_6 interactions with Sn(II) were also observed in tin(II) bis(diphenyl dithio-

Table I. Fractional Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Molecule 1[a]

atom	x/a	y/b	z/c	U_{eq}, Å^2
Sn1	0.08395 (3)	0.78180 (4)	0.11412 (3)	0.030
Sn2	−0.08869 (3)	0.64618 (4)	0.12391 (2)	0.030
Al1	−0.1011 (1)	0.8886 (2)	0.0470 (1)	0.030
Al2	0.0989 (2)	0.5325 (2)	0.1845 (1)	0.031
Cl1	−0.0284 (1)	0.7735 (2)	0.16694 (9)	0.033
Cl2	0.0226 (1)	0.6555 (1)	0.06914 (9)	0.032
Cl11	−0.1378 (1)	0.7599 (1)	0.0457 (1)	0.036
Cl12	0.0078 (1)	0.8853 (2)	0.0437 (1)	0.036
Cl13	−0.1306 (1)	0.9492 (2)	0.1151 (1)	0.044
Cl14	−0.1381 (2)	0.9475 (2)	−0.0196 (1)	0.044
Cl21	0.1298 (2)	0.4590 (2)	0.2476 (1)	0.045
Cl22	0.1376 (1)	0.6593 (2)	0.1909 (1)	0.042
Cl23	0.1303 (2)	0.4807 (2)	0.1134 (1)	0.045
Cl24	−0.0094 (1)	0.5416 (2)	0.1911 (1)	0.040
C10	0.3926 (7)	0.4901 (7)	0.1550 (5)	0.051
C11	0.4340 (7)	0.4541 (8)	0.1888 (5)	0.054
C12	0.4102 (7)	0.3914 (8)	0.2234 (4)	0.053
C13	0.3388 (8)	0.3750 (9)	0.2225 (5)	0.058
C14	0.2979 (7)	0.416 (1)	0.1870 (7)	0.061
C15	0.3239 (8)	0.4716 (9)	0.1533 (5)	0.062
C20	0.2778 (6)	0.3485 (7)	0.0452 (6)	0.043
C21	0.2464 (6)	0.2954 (9)	0.0765 (5)	0.048
C22	0.2647 (8)	0.2067 (9)	0.0732 (6)	0.061
C23	0.3082 (8)	0.1835 (7)	0.0366 (7)	0.051
C24	0.3368 (6)	0.244 (1)	0.0065 (5)	0.052
C25	0.3202 (8)	0.3223 (9)	0.0108 (6)	0.063
C30	0.1983 (6)	−0.0043 (9)	0.4363 (5)	0.054
C31	0.1523 (6)	0.0291 (7)	0.4706 (5)	0.044
C32	0.0892 (6)	0.0031 (7)	0.4681 (4)	0.044
C33	0.0667 (6)	−0.0523 (9)	0.4317 (5)	0.057
C34	0.1129 (8)	−0.0799 (8)	0.3955 (5)	0.061
C35	0.1836 (7)	−0.0550 (9)	0.3986 (6)	0.055
C40	0.2061 (7)	0.2555 (7)	0.3131 (6)	0.048
C41	0.2519 (7)	0.208 (1)	0.3412 (6)	0.064
C42	0.2597 (8)	0.129 (1)	0.3306 (9)	0.069
C43	0.223 (2)	0.092 (1)	0.292 (1)	0.075
C44	0.184 (1)	0.139 (2)	0.2644 (7)	0.071
C45	0.1728 (7)	0.217 (1)	0.2747 (6)	0.060
C50	0.4573 (7)	0.153 (1)	0.3406 (6)	0.064
C51	0.471 (1)	0.158 (1)	0.3922 (6)	0.077
C52	0.5121 (9)	0.213 (1)	0.4107 (6)	0.071
C53	0.5458 (7)	0.2693 (9)	0.3801 (9)	0.073
C54	0.5294 (9)	0.267 (1)	0.3272 (8)	0.070
C55	0.486 (1)	0.210 (1)	0.3094 (5)	0.074

[a] $U_{eq} = (U_1U_2U_3)^{1/3}$, with U_i being the eigenvalues of the $\mathbf{U}_{ij}$ matrix.

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Table II. Important Distances (Å) and Angles (deg) for Molecule 1[a]

	Bond Distances		
Sn1–Cl1	2.624 (2)	Sn2–Cl1	2.607 (2)
Sn1–Cl2	2.627 (2)	Sn2–Cl2	2.633 (2)
Sn1–Cl12	2.890 (3)	Sn2–Cl11	2.891 (2)
Sn1–Cl22	2.986 (3)	Sn2–Cl24	2.880 (3)
Sn1–D1	3.24	Sn2–D3	3.26
Sn1–D2	3.11	Sn2–D4	3.20
Al1–Cl11	2.177 (4)	Al2–Cl24	2.164 (4)
Al1–Cl12	2.167 (4)	Al2–Cl22	2.169 (4)
Al1–Cl13	2.098 (4)	Al2–Cl21	2.104 (4)
Al1–Cl14	2.100 (4)	Al2–Cl23	2.116 (4)
	Bond Angles		
Sn1–Cl1–Sn2	101.9 (1)	Sn1–Cl2–Sn2	101.1 (1)
Cl1–Sn1–Cl2	78.4 (1)	Cl1–Sn2–Cl2	78.6 (1)
Cl12–Sn1–Cl22	169.1 (1)	Cl11–Sn2–Cl24	166.5 (1)
D1–Sn1–D2	101.9	D3–Sn2–D4	99.7
Sn1–Cl12–Al1	120.7 (1)	Sn2–Cl11–Al1	117.8 (1)
Sn1–Cl22–Al2	115.6 (1)	Sn2–Cl24–Al2	122.3 (1)
interplane angles		C10–C15/C20–C25	66.3
		C30–C35/C40–C45	65.3
angle Sn–D/ring plane normal		C10–C15	8.8
		C20–C25	5.0
		C30–C35	10.4
		C40–C45	6.9

[a] D1–D4 are centroids of the respective benzene rings.

phosphate).[6] All these compounds have in common that only *one* arene molecule is attached to the group 14 metal center. In an attempt to obtain a first example of bis(arene) complexes, the reaction of $Sn(AlCl_4)_2$ with aromatic hydrocarbons was reinvestigated, and the results are presented in this communication.

Treatment of the freshly prepared molten salt $Sn(AlCl_4)_2$ with benzene under reflux conditions, followed by cooling the resulting solution, affords a benzene-containing colorless crystalline product, mp 110 °C, which is stable at room temperature. The crystal structure analysis[7] of this compound revealed the presence of a dimeric species of the composition $[(\eta^6\text{-}C_6H_6)_2SnCl(AlCl_4)]_2\cdot C_6H_6$ (1) with two η^6-coordinated benzene rings at each tin atom and one molecule of crystal benzene with no specific metal contacts (Figure 1). Two $(\eta^6\text{-}C_6H_6)_2Sn$ dications are bridged by two 1,3-bidentate tetrahedral $AlCl_4^-$ anions and by two two-coordinate Cl^- anions. The Sn(II) centers adopt a distorted octahedral coordination geometry. The structure can also be described as an eight-membered ring in an elongated chair form composed of Sn(1)–Cl(12)–Al(1)–Cl(11)–Sn(2)–Cl(24)–Al(2)–Cl(22) (Figure 2). The tin and the chlorine atoms of this ring are almost coplanar (maximum deviation from planarity 0.11 Å for Sn2), and the Al atoms lie above and below this plane, respectively (inclination angles of 116.17 and 112.43° with the planes Cl11–Al1–Cl12 and Cl22–Al2–Cl24, respectively). Although the molecule has no crystallographic symmetry, its structure approaches C_{2h} symmetry. Each tin atom is situated nearly centroid above its two arene rings, which are inclined with respect to each other, forming angles of 101.9° (Sn1) and 99.7° (Sn2), respectively. The angles between the normals to the arene planes and the Sn–D (see Table II) lines in the range between 5.0 and 10.4° are evidence for η^6-coordination. The observed very long distances of the Sn atoms from the ring centers (3.11 and 3.24 Å for Sn(1) and 3.26 and 3.20 Å for Sn(2)) indicate only very weak attractive forces between the two components. The Sn–Cl distances vary between 2.607 (2) and 2.633 (2) Å (Cl^- bridge) and 2.880 (3) and 2.986 (3) Å ($AlCl_4^-$ bridge). The packing of the molecules in the unit cell is such that there are no specific contacts between the individual dimers.

We note that there is an interesting parallel for the structure of 1 in one of the ylide complexes reported previously from this laboratory.[8] In $[(CH_3O)_3Ti(CH_2)_2P(CH_3)_2]_2$, the Ti(IV) are the centers of a similar pair of edge-sharing, doubly-chelated octahedra.

It is also noteworthy that the analogous reactions between freshly prepared $Sn(AlCl_4)_2$ melts and arenes of higher donor capability like hexamethylbenzene, xylene, or mesitylene do not lead to the formation of bis(arene) complexes. In the case of hexamethylbenzene, the tetrameric species $[(\eta^6\text{-}C_6Me_6)SnCl(AlCl_4)]_4$ was isolated (when chlorobenzene was used as a solvent),[5] whereas no crystalline arene complexes were obtained with xylene and mesitylene.[9] With lead as the central atom and benzene as the donor component, the monoarene complex $(\eta^6\text{-}C_6H_6)Pb(AlCl_4)_2\cdot C_6H_6$ was isolated.[4b] In the case of the corresponding $Pb(AlBr_4)_2$ system, benzene-containing crystals were formed, which were shown to be composed of cross-linked $Pb(AlBr_4)_2$ units without any metal–arene contacts.[5]

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Supplementary Material Available: Tables of atomic coordinates, anisotropic thermal parameters, and hydrogen atom coordinates (7 pages); a listing of structural factors (31 pages). Ordering information is given on any current masthead page.

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(7) *Crystal structure data* for $1\cdot C_6H_6$: Syntex $P2_1$ diffractometer; Mo Kα radiation; λ = 0.71069 Å; graphite monochromator; T = −50 °C; $C_{30}H_{30}Al_2Cl_{10}Sn_2$, M_r = 1036.477; orthorhombic, space group *Pbca* (No. 61); a = 19.869 (1), b = 15.941 (1), c = 25.954 (2) Å; V = 8220.5 Å^3; Z = 8; d_{calcd} = 1.675 g/cm^3; μ(Mo Kα) = 19.4 cm^{-1}; $F(000)$ = 4048; 7144 independent reflections were measured, 5542 of which with $F_o \geqslant 4.0\,\sigma(F_o)$ were deemed "observed" and used for all further calculations (*hkl*, +23, +18, +30; $((\sin\theta/\lambda)_{max}$ = 0.595 Å^{-1}, ω scan, $\Delta\omega$ = 0.8°). Lp and empirical absorption corrections were applied to the data. Solution was by Patterson methods (SHELXS-86). Anisotropic refinement (H atoms constant with U_{iso} = 0.05 Å^2) converged at R (R_w) = 0.052 (0.072) (397 refined parameters, function minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F_o)$, SHELX-76). The final difference map was featureless with $\Delta\rho_{max/min}$ = +0.82/−0.95 e/Å^3.

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When Phenylcyclopropanes Are Generated by γ-Ionization of *erythro*- and *threo*-d_2-$C_5H_5(CO)_2FeCHDCHDCH(OCH_3)C_6H_5$, Cleavage of the Fe–$C_\alpha$ Bond Occurs with Inversion of Configuration of C_α

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Summary: 2,3-Dideuterio-r-1-phenylcyclopropanes are stereospecifically generated by abstraction of the γ-methoxide groups from *erythro*- and *threo*-d_2-$C_5H_5(CO)_2Fe$–CHD–$CHDCH(OCH_3)C_6H_5$ using TMSOTf. These results established that the cyclopropane ring is formed by backside attack of electrophilic C_γ on C_α with net inversion of stereochemistry at C_α.

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