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1,1,12,12-Tetra-*n*-butyl[1.1]stannaferrocenophane: Preparation and Crystal and Molecular Structure

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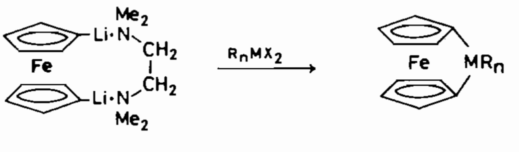
The compound 1,1,12,12-tetra-n-butyl[1.1] stannaferrocenophane, $C_{36}H_{52}Fe_2Sn_2$, was obtained from the reaction of 1,1'-dilithioferrocene-tetramethylethylenediamine with di-n-butyltin dichloride. The crystal is triclinic with $a = 10.217(3)$, $b = 10.748(4)$, $c = 10.173(3)$ Å, $\alpha = 116.34(3)$, $\beta = 90.21(2)$, $\gamma = 116.35(2)°$, $V = 868.7$ Å^3. The structure was solved by conventional three-dimensional Patterson heavy-atom methods using 4013 unique reflections having intensities greater than 3σ. Refinement was carried out by a full-matrix least-squares technique to a final R of 0.046. The molecule consists of two ferrocene moieties linked to each other through tin atoms. Bond distances and angles are regular. One butyl group on each tin atom is disordered.

When the 1,1'-dilithioferrocene-tetramethylethylene-diamine complex, *1*, reacts with dihalides of silicon, germanium phosphorus and arsenic, [1]-ferrocenophanes, *2*, result [1–3]. Dimethyl- and diphenyltin dichlorides are exceptions. On reaction of these with *1* in THF at –78 °C only orange, resinous, products of higher molecular weight were produced [2].



(1)

1 *2*

Since our more successful preparations of (1,1'-ferrocenediyl)organophosphines had been carried out in hexane rather than in tetrahydrofuran [3], we examined the reaction of *1* with di-*n*-butyltin dichloride using hexane as the reaction solvent.

Experimental

Synthesis

A 300 ml three-necked Morton (creased) flask was charged with 4.00 g (12.6 mmol) of 1,1'-dilithioferrocene·TMEDA [4] in a Vacuum Atmospheres glovebox. The flask, which was fitted with a gas inlet tube and two no-air stoppers, then was removed from the glovebox and attached to a Schlenk manifold. The no-air stopper of the middle neck was replaced with a mechanical stirring unit against a counter-flow of nitrogen. Hexane (200 ml) was cannulated into the flask and the yellow slurry was cooled to –78 °C while stirring.

Meanwhile, a flame-dried, nitrogen-purged 50 ml one-necked, round-bottomed flask was charged with 3.95 g (13.0 mmol) of di-*n*-butyltin dichloride (M & T Chemicals, recrystallized from hexane) and 25 ml of hexane. The resulting solution was cooled to –78 °C and then was cannulated into the dilithioferrocene slurry. The reaction mixture then was stirred vigorously while it was allowed to warm slowly. No color changes were observed and the reaction mixture was stirred at room temperature overnight. It then was treated with 10 ml of saturated aqueous NH_4Cl. The mixture was filtered under nitrogen and the filtrate was evaporated under reduced pressure. Filtration chromatography of the residue (100 mesh silicic acid, Mallinckrodt reagent grade: 15% CH_2Cl_2/85% hexane) removed an orange band which gave 0.46 g of golden solid upon removal of solvent. Recrystallization from hexane at –30 °C yielded 0.204 g (3% yield) of golden nuggets of 1,1,12,12-tetra-*n*-butyl[1.1]stannaferrocenophane, mp 140–141 °C. *Anal.* Calcd. for $C_{36}H_{52}Sn_2Fe_2$: C, 51.84; H, 6.30. Found: C, 51.91; H, 6.23. *Mass Spectrum* (m/z, relative intensity, %): 834 (M^+, 31.9), 777 ($M^+ - 1C_4H_9$, 100.0), 663 ($M^+ - 3C_4H_9$, 37.5), 606 ($M^+ - 4C_4H_9$, 47.2), 486 ($M^+ - 4C_4H_9 - Sn$, 26.8) for mass range 850–400 m/z.

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IR Spectrum (CH_2Cl_2): 3100(w), 2970(s), 2939(s), 2883(m), 2862(m), 1463(m), 1378(m), 1297(w), 1185(w), 1139(s), 840(m), 826(m), 660(w), 593(w), cm^{-1}. *^1H NMR spectrum* ($CDCl_3$): δ 0.73–1.60 (m, 18H, C_4H_9, with $^{117,119}Sn$ satellites), 4.15 (t, J = 1.8 Hz, 4H, Cp) and 4.37 ppm (t, J = 1.8 Hz, 4H, Cp). *UV-vis Spectrum* (CH_2Cl_2): $\lambda = 465$ ($\epsilon = 174$ cm^2 mol^{-1}) and 332 nm ($\epsilon = 90$ cm^2 mol^{-1}).

A similar reaction between 1,1′-dilithioferrocene and di-*n*-butyltin dichloride which was carried out using 1,2-dimethoxyethane as reaction medium gave only polymeric products. In both the hexane and the 1,2-dimethoxyethane reactions these, tan, insoluble products were not examined further, although, in principle, they should provide a preparative entry to functional ferrocene derivatives *via* electrophilic Sn–C cleavage reactions.

X-ray Data

A prism shaped crystal was mounted so that its long axis was roughly parallel to the ϕ axis of a CAD-4 automated diffractometer. Preliminary examination and data collection were performed with incident beam monochromatized (graphite crystal) MoKα radiation ($\lambda = 0.71073$ Å).

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 high-angle reflections measured by the computer controlled diagonal slit method of centering. The triclinic cell parameters and calculated volume are: $a = 10.217(3)$, $b = 10.748(4)$, $c = 10.173(3)$ Å, $\alpha = 116.34(3)$, $\beta = 90.21(2)$, $\gamma = 116.35(2)°$, $V = 868.7$ Å^3. For $Z = 1$ and F.W. = 833.89 the calculated density is 1.59 g/cm^3. As a check on crystal quality, omega scans of several intense reflections were measured; the width at half-weight was 0.20° with a take-off angle of 2.8°, indicating good crystal quality. There were no systematic absences; the space group was determined to be $P\bar{1}$ by the structure solution.

The data were collected at a temperature of 23 ± 1° using the omega-theta scan technique. The scan rate varied from 2 to 20°/min (in omega). Data were collected to a maximum two-theta value of 60.0°. The scan range (in degrees) varied according to the formula $\theta = 0.7 + 0.347 \tan \theta$. Moving crystal moving-counter background counts were made by scanning an additional 25% above and below this range. The counter aperture was also adjusted as a function of θ. The horizontal aperture width ranged from 2.0 to 3.0 mm; the vertical aperture was set at 2.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21 cm. For intense reflections an attenuator was automatically inserted in front of the detector; the attenuator factor was 20.7. A total of 5216 reflections was collected, of which 5050 were unique. As a check on crystal and electronic stability 3 representative reflections were measured every 40 min. A decay correction was applied. The correction factors on I ranged from 0.95 to 1.11 with an average value of 1.03. Reflection intensities were calculated as $I = S(C - RB)$ where S is the scan rate, C is the total integrated peak count, B is the total background count and R the ratio of scan time for the peak to the scan time for the background *i.e.*, 2. The new intensity data were corrected for Lorentz-polarization effects, but not for absorption (μ (MoKα) = 22.9 cm^{-1}). An extinction correction was not necessary.

Structure Solution and Refinement

The structure was solved by the Patterson heavy atom method which revealed the positions of the Sn and Fe atoms symmetrically placed about a center of symmetry (Fig. 1). The remaining atoms were located in succeeding difference Fourier synthesis. One of the n-butyl groups is disordered, with two orientations for the -$CH_2CH_2CH_3$ moiety. Atoms C16–C17–C18 and C19–C20–C21 define the two orientations (see Fig. 1) and were all refined at 50%

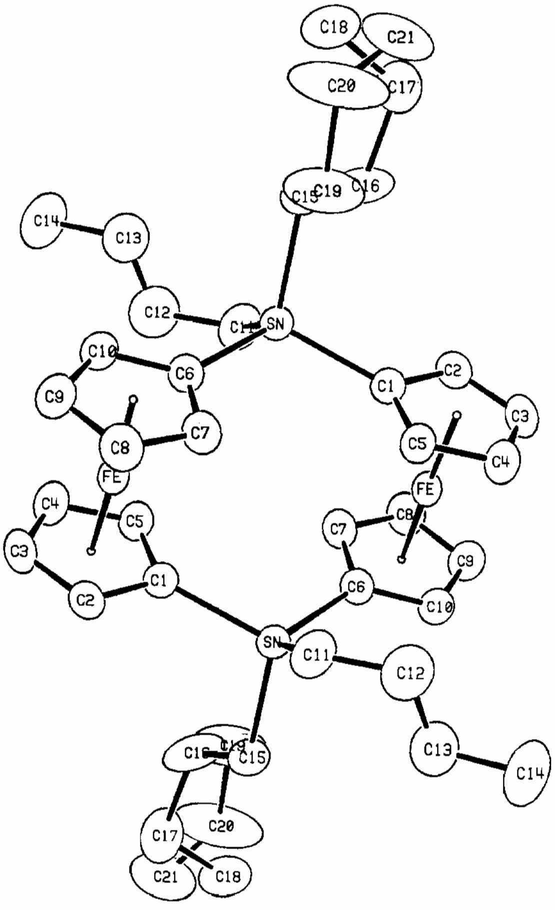


Fig. 1. Schematic representation of the dibutyldiferrocenyltin(IV) complex with the numbering scheme used in the Tables.

occupancy. The disorder scheme is shown in Figure 1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were not included in the calculations. The structure was refined by full-matrix least-squares procedures; where the function minimized was $\Sigma w(|F_o| - |F_c|)^2$ and the weights w were equal to $4F_o^2/\sigma^2(F_o^2)$ or $4F_o^2/[\sigma(F_o^2)]^2$.

The standard deviation on intensities, $\sigma(F_o^2)$, is defined as follows

$$\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/(Lp)^2$$

where Lp is the Lorentz-polarization factor, and the parameter p is a factor introduced to downweight intense reflections. Here p was set to 0.050. Scattering factors were taken from Cromer and Waber [5]. Anomalous dispersion effects were included in the calculation of the structure factors [6]; the values for $\Delta f'$ and $\Delta f''$ were those of Cromer [7]. Only the 4013 reflections having intensities greater than 3.0 times their standard deviation were used in the refinements. The final cycle of refinement included 208 variable parameters and converged (largest parameter shift was 0.03 times its esd) with unweighted and weighted agreement factors of:

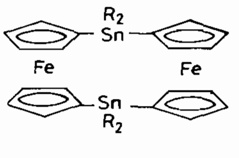
$$R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.046$$

$$R2 = (\Sigma w(|F_o| - |F_c|)^2/\Sigma wF_o^2)^{1/2} = 0.071$$

The standard deviation of an observation of unit weight was 2.00 [8]. The highest peak in the final difference Fourier was in the region of the Sn atoms and is presumably due to absorption errors. Plots of $\Sigma w(|F_o| - |F_c|)^2$ versus $|F_o|$, reflection order in data collection, sin θ/λ, and various classes of indices showed no unusual trends.

Results and Discussion

In agreement with a previous report [2], the reaction of 1,1'-dilithioferrocene with di-*n*-butyltin dichloride gave mostly insoluble, obviously polymeric products. However, a low yield of a soluble product could also be isolated. Although its C/H analysis indicated a $Fe(C_5H_4-)_2Sn(C_4H_9)_2$ stoichiometry, its mass spectrum showed a molecular ion equivalent to a dimeric species. Also, its UV-vis spectrum did not show the shifts characteristic for [1]ferrocenophanes [2,3]. On the basis of this evidence, the soluble product was *3*, rather than *2* (R = *n*-C_4H_9).



(R = n - C_4H_9)

3

This structure assignment was confirmed by an X-ray diffraction study.

Table I lists the positional and thermal parameters for all the non-hydrogen atoms. Figure 1 gives a schematic view of the structure with the numbering scheme used in the tables. Figure 2 presents the same view as the previous figure but with only one of each of the disordered butyl groups per tin atom shown. A structure factor table is available as supplementary material [12]. Interatomic distances are collected in Table II and interatomic angles in Table III.

The molecule consists of two ferrocene moieties linked to each other through tin atoms bonded to both the upper and lower five membered rings. Each tin atom is tetrahedrally coordinated by carbon atoms from two butyl groups and two cyclopentadienyl rings. The molecule itself possesses a center of inversion. The Sn–C bond distances are normal with those to the butyl groups being slightly larger as might be expected for sp^3 hybridized carbon atoms.

All of the iron–carbon bond distances in the ferrocene moieties are equal within experimental error. The average Fe–C distance, 2.045(8) Å, compares well with that in bis-(fulvalene)diiron, 2.043(6) Å, and other ferrocene compounds in which the rings are anchored [9, 10]. This is also the case for the Fe-cyclopentadienyl ring centroid distances. The carbon–carbon distances in the C5 rings are somewhat longer than is normally observed [11] the average value being 1.430 Å. This is undoubtly due to the reduced thermal motion of the rings resulting from their being bonded to the Sn atoms. In bis(fulvalene) diiron, where the rings are also restricted in their thermal motion, the average C–C ring distance is 1.436 Å with a similar range in values.

Each of the cyclopentadienyl rings is perfectly planar as seen from the χ^2 values and deviations of ring atoms from the plane (Table IV). The rings are very nearly in the eclipsed conformation as shown by the ring 1-C-Fe-C-Fe-ring 2 interatomic angles. The dihedral angle between the rings in each ferrocene group is 2.5°. There is a slight twisting of the ferrocene groups about the center of inversion as shown by the values of the torsion angles listed in Table III. This twisting is also evident in the deviation of the atoms from the mean plane which includes the C1 to C5 atoms of one ferrocene group.

One butyl group on each of the tin atoms is disordered. Two sets of positions for the carbon atoms of the disordered group were found and refined at 50% occupancy. Both the disordered butyl groups contain one long and two short C–C bonds which must result from the difficulty of placing these atoms with any degree of accuracy.

Finally, the difference in the results observed in the case of diorganotin dichlorides *vs.* diorganosilicon

TABLE I. Table of Positional and Thermal Parameters and Their Estimated Standard Deviations.[a]

Atom	*x*	*y*	*z*	*B(1, 1)*	*B(2, 2)*	*B(3, 3)*	*B(1, 2)*	*B(1, 3)*	*B(2, 3)*	*Beqv*
SN	0.46606(3)	0.36321(3)	0.16709(3)	4.374(9)	3.265(6)	3.766(8)	2.244(5)	1.564(7)	1.945(5)	3.562(6)
FE	0.42692(6)	0.71511(5)	0.20777(6)	3.25(2)	2.44(1)	3.13(2)	1.55(1)	0.99(1)	1.10(1)	3.01(1)
C1	0.3544(5)	0.4958(4)	0.1927(5)	3.9(1)	2.84(9)	4.2(1)	1.67(8)	1.4(1)	1.81(8)	3.59(9)
C2	0.3542(5)	0.6106(5)	0.3393(5)	5.1(2)	3.8(1)	4.0(1)	2.49(9)	2.1(1)	2.02(9)	4.1(1)
C3	0.2595(5)	0.6675(5)	0.3168(5)	4.7(1)	4.1(1)	5.3(2)	2.6(1)	2.8(1)	2.3(1)	4.5(1)
C4	0.1967(5)	0.5893(5)	0.1595(6)	3.5(1)	3.6(1)	5.6(2)	1.91(9)	1.4(1)	2.0(1)	4.3(1)
C5	0.2549(4)	0.4830(4)	0.0824(5)	3.3(1)	2.8(1)	4.6(1)	1.40(8)	1.1(1)	1.59(9)	3.7(1)
C6	0.4493(5)	0.2224(4)	−0.0683(5)	4.1(1)	2.91(9)	3.5(1)	1.74(8)	1.2(1)	1.33(8)	3.61(9)
C7	0.3541(5)	0.1952(5)	−0.1939(5)	3.6(1)	3.4(1)	4.3(2)	1.48(9)	1.2(1)	1.6(1)	4.0(1)
C8	0.3667(5)	0.0858(5)	−0.3309(6)	3.9(2)	3.3(1)	4.3(2)	1.2(1)	0.7(1)	1.1(1)	4.5(1)
C9	0.4707(5)	0.0444(4)	−0.2919(5)	5.2(2)	2.5(1)	4.7(2)	1.90(9)	1.3(1)	1.2(1)	4.4(1)
C10	0.5180(5)	0.1275(4)	−0.1299(5)	5.3(2)	3.09(9)	4.6(1)	2.41(9)	1.8(1)	1.94(9)	4.1(1)
C11	0.6942(6)	0.5215(6)	0.3054(6)	5.2(2)	5.4(1)	5.2(2)	3.0(1)	0.1(2)	2.1(1)	5.4(1)
C12	0.8094(8)	0.4642(8)	0.2672(9)	5.8(2)	8.7(2)	9.3(3)	3.8(2)	1.6(2)	5.1(2)	7.6(2)
C13	0.7774(8)	0.3317(8)	0.2807(9)	7.4(3)	7.9(2)	9.6(3)	4.7(2)	3.6(2)	5.2(2)	7.6(2)
C14	0.8984(8)	0.2830(8)	0.2580(12)	9.9(3)	11.5(2)	18.2(6)	8.4(2)	7.6(3)	9.9(3)	10.8(3)
C15	0.3383(6)	0.1990(5)	0.2464(6)	5.4(2)	4.9(1)	5.7(2)	2.9(1)	2.1(1)	3.48(9)	4.9(1)
C16	0.1728(12)	0.1353(12)	0.2058(14)	3.7(4)	10.6(3)	16.7(4)	2.9(2)	3.3(3)	11.8(2)	8.4(3)
C17	0.0888(14)	0.0523(13)	0.3019(18)	6.3(4)	6.0(3)	14(2(7)	3.4(3)	5.8(4)	6.4(3)	8.0(4)
C18	0.0852(16)	−0.1032(12)	0.2423(14)	8.1(6)	4.6(3)	7.4(5)	2.4(3)	1.7(5)	3.7(3)	6.8(4)
C19	0.1953(21)	0.0945(17)	0.1522(17)	8.7(9)	8.3(5)	8.8(5)	0.2(6)	0.1(6)	6.2(3)	9.5(5)
C20	0.0860(27)	−0.0530(19)	0.1896(18)	13(1)	7.9(5)	9.0(6)	−2.6(7)	−0.4(8)	6.0(3)	12.7(7)
C21	0.0649(18)	−0.0035(17)	0.3342(16)	7.3(7)	10.4(6)	11.8(6)	1.8(6)	4.2(5)	8.4(3)	9.7(5)

Atom	*x*	*y*	*z*		*Atom*	*x*	*y*	*z*	*B, A^2*
CP1	0.2839	0.5692	0.2181		CP2	0.4317	0.1350	−0.2030	

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[a] The form of the anisotropic thermal parameter is: exp[$-0.25Ch^2a^2B(1,1) + k^2b^2B(2,2) + l^2c^2B(3,3) + 2hkabB(1,2) + 2hlacB(1,3) + 2klbcB(2,3)$]] where a, b, and c are reciprocal lattice constants. Estimated standard deviations in the least significant digits are shown in parentheses. 'Atoms' CP1 and CP2 are the centroids of cyclopentadienyl rings one and two respectively. Atoms C16–C21 were refined at 50% occupancy.

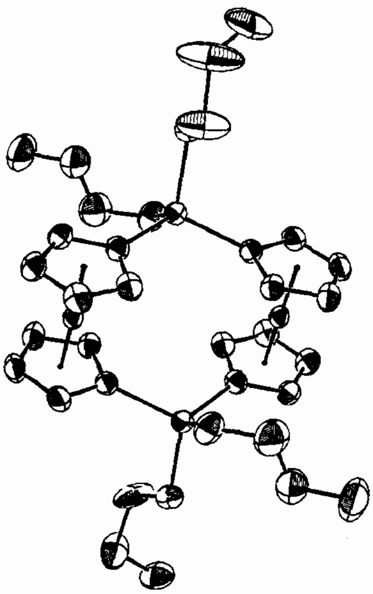


Fig. 2. Schematic representation of the dibutyldiferrocenyltin(IV complex showing the thermal ellipsoids (at the 50% probability level) but with only one of each of the disordered butyl groups.

TABLE II. Table of Bond Distances in Angstroms.[a]

Sn–C1	2.125(3)	C1–C5	1.429(5)
Sn–C6	2.146(3)	C2–C3	1.418(6)
Sn–C11	2.154(4)	C3–C4	1.414(6)
Sn–C15	2.167(4)	C4–C5	1.444(5)
Fe–C1	2.063(3)	C6–C7	1.439(5)
Fe–C2	2.051(4)	C6–C10	1.414(5)
Fe–C3	2.039(4)	C7–C8	1.423(5)
Fe–C4	2.044(4)	C8–C9	1.435(6)
Fe–C5	2.051(3)	C9–C10	1.432(5)
Fe–C6	2.042(3)	C11–C12	1.534(7)
Fe–C7	2.041(3)	C12–C13	1.383(9)
Fe–C8	2.047(4)	C13–C14	1.524(8)
Fe–C9	2.035(3)	C15–C16	1.49(2)
Fe–C10	2.038(4)	C15–C19	1.38(3)
Fe–CP1	1.649	C16–C17	1.61(3)
Fe–CP2	1.639	C17–C18	1.49(2)
C1–C2	1.455(5)	C19–C20	1.69(3)
		C20–C21	1.38(3)

[a]Numbers in parentheses are estimated standard deviations in the least significant digits. CP1 and CP2 are cyclopentadienyl ring centroids.

TABLE III. Important Interatomic and Torsion Angles (deg.).

Interatomic Angles

C1–Sn–C6	110.0(1)	Sn–C1–C2	123.3(3)
C1–Sn–C11	110.0(2)	Sn–C1–C5	130.4(2)
C1–Sn–C15	104.9(1)	Sn–C6–C7	126.0(3)
C6–Sn–C11	114.0(2)	Sn–C6–C10	126.8(3)
C6–Sn–C15	107.4(2)	Sn–C11–C12	118.4(4)
C11–Sn–C15	110.1(2)	Sn–C15–C16	113.6(8)
		Sn–C15–C19	109.(1)
C1–Fe–C7	108.4(1)	C6–Fe–C5	108.9(1)
C1–Fe–C6	121.5(1)		
C1–Fe–C8	125.1(2)	C6–Fe–C4	125.8(2)
C3–Fe–C9	104.5(2)	C8–Fe–C2	106.5(2)
C3–Fe–C8	118.3(2)		
C3–Fe–C10	123.8(2)	C8–Fe–C1	125.1(2)
CP1–Fe–CP2	177.9		
C1–C2–C3	109.0(3)	C6–C7–C8	108.7(3)
C2–C3–C4	108.3(3)	C7–C8–C9	107.8(3)
C3–C4–C5	107.9(3)	C8–C9–C10	107.1(3)
C4–C5–C1	108.7(3)	V9–C10–C6	109.4(3)
C5–C1–C2	106.0(3)	C10–C6–C9	106.9(3)
C11–C12–C13	114.6(5)	C15–C16–C17	111(2)
C12–C13–C14	115.2(6)	C16–C17–C18	109(2)
C15–C19–C20	115(2)	C19–C20–C21	116(2)

Torsion Angles

C1–Sn–C6–Fe	+81.5
C6–Sn–C1–Fe	−85.6
C1–Sn–C6–C7	−11.0
C6–Sn–C1–C5	+11.9
C1–Sn–C6–C10	+175.2
C6–Sn–C1–C2	−175.9

TABLE IV. Least Squares Planes and Deviations (Å) of Atoms from Planes.

a. C1–C5 ring $x^2 = 5$

$0.4693x + 0.8812y - 0.0564z - 3.8342 = 0$

C1	0.005(5)	C4	−0.001(5)
C2	−0.008(5)	C5	−0.003(5)
C3	0.006(6)	Sn	−0.160

b. C6–C10 ring $x^2 = 8$

$0.4421x + 0.8966y - 0.0255z - 3.8011 = 0$

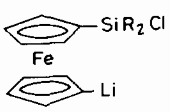
C6	0.006(5)	C9	+0.008(5)
C7	−0.002(5)	C10	−0.009(5)
C8	−0.005(6)	Sn	−0.123

c. C1–C5 ring + Sn $x^2 = 338$

$0.5090x + 0.8686y - 0.0613z - 3.7267 = 0$

Sn	0.000	C3	−0.031(5)
C1	0.068(5)	C4	−0.043(5)
C2	0.019(5)	C5	0.017(5)
C6	0.244(5)	C9	0.411(5)
C7	0.206(5)	C10	0.348(5)
C8	0.303(6)	C11	0.433(6)
		C15	−2.012(6)

and diorganogermanium dichlorides merits consideration. It is known [13] that monochlorosilanes react rather slowly with lithium reagents, especially at low temperature, whereas monochlorostannanes react rapidly under the same conditions. Consider, then, the first intermediates in each case, *4* and *5*. The relatively low reactivity of the Si–Cl bond in *4* will favor the observed selectivity for intramolecular reaction. On the other hand, the more reactive



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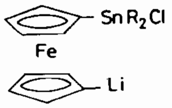

5

Sn–Cl bond in *5* is less discriminating, and, since the opportunities for *inter*molecular reaction are much greater, will form dimers and, to a much greater extent, higher oligomers and polymeric products.

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