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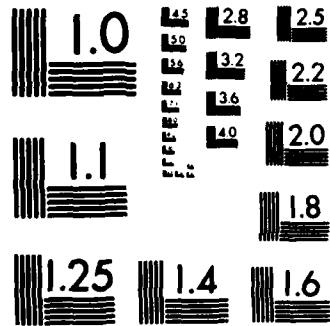
MACROCYCLES CONTAINING TIN SYNTHESIS AND STRUCTURE OF
110-DIPHENYL-110-DI... (U) TEXAS A AND M UNIV COLLEGE
STATION DEPT OF CHEMISTRY M NEWCOMB ET AL. 02 JUL 84
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TECHNICAL REPORT NO. 5

Macrocycles Containing Tin.

Synthesis and Structure of

1,10-Diphenyl-1,10-distannabicyclo[8.8.8]hexacosane

by

Martin Newcomb, Michael T. Blanda, Yutaka Azuma, and Terry J. Delord

Texas A&M University
Department of Chemistry
College Station, TX 77843

July 2, 1984

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Macrocycles Containing Tin. Synthesis and Structure of
1,10-Diphenyl-1,10-distannabicyclo[8.8.8]hexacosane

Martin Newcomb*†, Michael T. Blanda, Yutaka Azuma, and Terry J. Delord
Department of Chemistry, Texas A&M University, College Station, TX, 77843 USA

Summary: The title macrobicyclic compound was prepared and studied by x-ray crystallography.

We have reported the syntheses of macrocycles containing two or four diphenylstanna moieties in the ring with various length polymethylene chains between the tin atoms.¹ Since acyclic, Lewis acidic distanna compounds have been shown to exhibit cooperative binding of donors,² we predicted that macrocycles containing Lewis acidic tin atoms would display cooperative and size-selective binding properties. In confirmation of this expectation, we have found that replacement of one or both phenyl groups on each tin atom in a macrocycle with chlorine or bromine atoms gives Lewis acidic macrocycles which form complexes with donor compounds containing basic nitrogen and oxygen atoms; complexes with donor atom to tin atom ratios of both 2:1 and 1:1 have been isolated and are under investigation.³ In this communication we report the preparation and structure of a representative of a new class of organotin compounds, a macrobicyclic compound containing two tin atoms in which the tin atoms serve as bridgeheads in a manner analogous to the nitrogen atoms of a cryptand. It may be expected that Lewis acidic macrobicyclic organotin compounds will be highly selective complexing agents.

The preparation of 1,10-diphenyl-1,10-distannabicyclo[8.8.8]hexacosane (1) follows the sequence shown in the Scheme. The preparation of macrocycle 4 has been reported,¹ but we modified the procedure by using a double dilution method wherein dilute (ca. 0.03 M) solutions of the reagents 2 and 3¹ were added

simultaneously to a THF reaction mixture over 2 h at 25 °C to give **4** in 62% yield after purification by reverse phase chromatography¹ and recrystallization. Treatment of macrocycle **4** with 2.1 molar equivalents of HCl in CH₂Cl₂ at -78 °C followed by slow warming to 25 °C and distillation of the solvent at reduced pressure gave the dichlorinated product **5** which appeared to be pure by ¹H NMR spectroscopy. Crude **5** was then allowed to react with the diGrignard reagent **2** in THF at 25 °C, again using the double dilution method (0.03 M solutions, 2 h addition). After a standard workup,¹ bicycle **1** was purified by preparative reverse phase chromatography (Baker bonded-phase ODS, 40 μ; 60:40, CH₃OH:THF elution; ca. 5 column volume retention) followed by recrystallization from CH₃OH/THF to give pure **1** in 15% yield from macrocycle **4**. Macrobicycle **1** (mp 92-94 °C) was characterized by ¹H NMR and ¹³C NMR spectroscopy, osmometric MW measurement (Calcd: 728; found: 734), and elemental analysis (C, H).

A crystal of **1** was suitable for x-ray crystallographic analysis.[†] An ORTEP drawing of **1** is shown in Figure 1. The compound crystallized in a cylindrical form with a tin-tin distance of 8.45 Å. The interior of the cavity in **1** is filled with the hydrogen atoms of the polymethylene chains. The tin atoms of **1** are essentially tetrahedral in the crystal, and from the ¹J(¹¹⁹Sn-¹³C) of 332 Hz appear to be tetrahedral in solution.⁴

Crystal data: Sn₂C₃₆H₅₈, M = 728.24, triclinic, space group $\bar{P}1$, a = 13.508(8), b = 16.154(9), c = 9.094(3) Å, α = 106.35(4)°, β = 109.67(4)°, γ = 92.49(5)°, U = 1772(1) Å³, Z = 2, D_c = 1.365 g/cm³. Intensity data were collected on an Enraf-Nonius CAD-4 diffractometer with Mo-Kα radiation. Data were corrected for Lorentz, polarization and absorption effects (μ = 14.38 cm⁻¹) to yield 2193 observed structure factors with I > 3σ(I) which were used for the structure solution and refinement. The positions of the two tin atoms were determined by direct methods. Successive least squares refinement and difference-Fourier methods were used to

locate the remaining carbon atoms. While exhibiting rather high thermal motion, all non-hydrogen atoms were successfully refined anisotropically. The hydrogen atoms were included in calculated positions with fixed temperature factors and were not refined. Least squares refinement converged at $R = 0.061$ and $R_w = 0.084$.

Other size distanna macrobicycles should be available by the procedures we used to prepare **1**, and, indeed, the corresponding 32 atom bicycle has already been prepared in our laboratory. In addition, functionalization of macrobicycle **1** to give a Lewis acidic species (replacement of phenyl groups with chloro groups) was achieved by treatment of **1** with HCl in a reaction similar to that used for conversion of **4** to **5**, and complexation properties of this derivative are under investigation.³

We are grateful for financial support from the Office of Naval Research.

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2. (a) T. J. Karol, J. P. Hutchinson, J. R. Hyde, H. G. Kuivila, and J. A. Zubieta, Organometallics, 1983, **2**, 106. (b) K. Jurkschat and M. Gielen, Bull. Soc. Chim. Belg., 1982, **91**, 803.
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4. H. G. Kuivila, J. L. Considine, R. H. Sarma, R. J. Mynott, J. Organometal. Chem., 1976, **111**, 179.

†Camille and Henry Dreyfus Teacher-Scholar, 1980-1985.

‡All crystallographic computing and plotting was performed using the Enraf-Nonius Structure Determination Package (1982), Enraf-Nonius, Delft, Holland. The atomic coordinates for this work are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Rd., Cambridge CB2 1EW. Any request should be accompanied by the full literature citation for this communication.

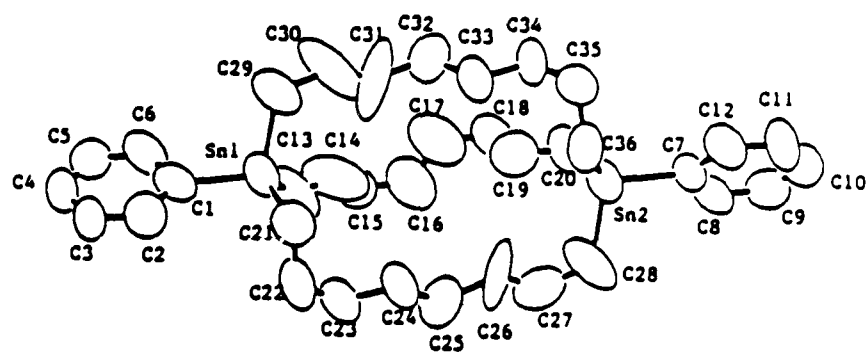


Figure 1. ORTEP representation of L. The hydrogen atoms have been omitted for clarity.

Scheme

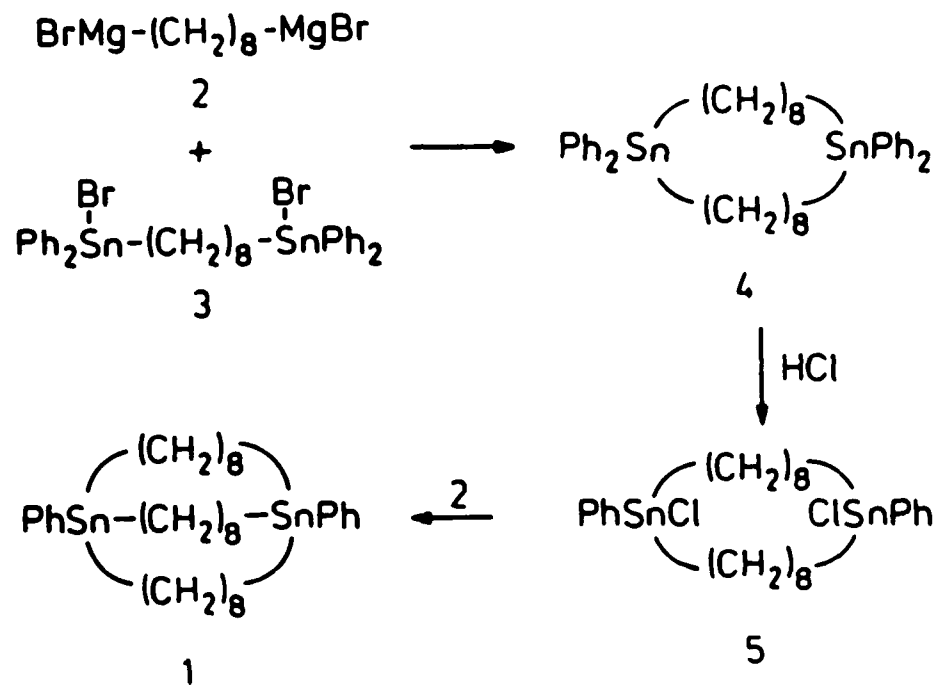


Table of Positional and Thermal Parameters and Their Estimated Standard Deviations.

Atom	x	y	z	A(1,1)	A(2,2)	A(3,3)	A(1,2)	A(1,3)	A(2,3)
SM1	0.32226(9)	0.52545(6)	0.1821(1)	0.0180(1)	0.00689(5)	0.0316(2)	0.0083(1)	0.0167(2)	0.0086(2)
SM2	0.17781(8)	-0.02537(6)	-0.1653(1)	0.0182(1)	0.00780(5)	0.0392(2)	0.0086(1)	0.0273(2)	0.0138(1)
C1	0.339(1)	0.6557(8)	0.259(1)	0.014(1)	0.0098(7)	0.038(2)	0.010(1)	0.028(2)	0.017(2)
C2	0.293(1)	0.7121(9)	0.359(1)	0.018(1)	0.0093(8)	0.038(2)	0.0094(2)	0.032(2)	0.017(2)
C3	0.306(1)	0.8024(9)	0.405(2)	0.017(1)	0.0059(7)	0.055(4)	0.002(2)	0.026(3)	0.001(3)
C4	0.367(1)	0.8494(8)	0.363(2)	0.017(2)	0.0048(6)	0.061(4)	0.003(2)	0.018(4)	0.012(2)
C5	0.412(1)	0.8943(8)	0.263(2)	0.014(1)	0.0091(7)	0.054(3)	0.004(2)	0.018(3)	0.026(2)
C6	0.404(1)	0.7141(9)	0.210(1)	0.019(1)	0.0185(8)	0.029(2)	0.013(2)	0.027(2)	0.014(2)
C7	0.162(1)	-0.1632(8)	-0.245(1)	0.013(1)	0.0058(6)	0.034(2)	0.005(1)	0.013(3)	0.006(2)
C8	0.096(1)	-0.2136(8)	-0.407(1)	0.015(1)	0.0095(7)	0.037(2)	0.012(2)	0.025(3)	0.019(2)
C9	0.087(1)	-0.3028(9)	-0.458(2)	0.014(1)	0.0091(9)	0.035(3)	0.003(2)	0.014(3)	0.000(3)
C10	0.133(1)	-0.3459(9)	-0.355(2)	0.017(1)	0.0072(8)	0.041(3)	0.004(2)	0.020(3)	0.000(3)
C11	0.193(1)	-0.3007(8)	-0.201(2)	0.023(2)	0.0068(6)	0.043(3)	0.006(2)	0.015(4)	0.014(2)
C12	0.210(1)	-0.2109(9)	-0.147(2)	0.016(2)	0.0077(8)	0.038(3)	0.006(2)	0.001(3)	0.003(2)
C13	0.256(2)	0.4763(12)	-0.077(2)	0.031(2)	0.0153(12)	0.054(4)	0.027(2)	0.043(4)	0.022(3)
C14	0.265(2)	0.4040(15)	-0.170(2)	0.021(2)	0.0254(20)	0.035(4)	0.023(3)	0.008(4)	0.007(4)
C15	0.225(1)	0.3698(11)	-0.353(2)	0.017(1)	0.0125(11)	0.029(3)	0.006(2)	0.013(3)	0.008(3)
C16	0.209(1)	0.2717(11)	-0.424(2)	0.028(2)	0.0145(11)	0.038(3)	0.015(3)	0.033(4)	0.022(3)
C17	0.294(1)	0.2248(12)	-0.403(2)	0.030(2)	0.0193(14)	0.042(3)	0.023(3)	0.046(3)	0.029(3)
C18	0.279(1)	0.1298(10)	-0.445(2)	0.021(1)	0.0112(9)	0.050(3)	0.013(2)	0.039(3)	0.020(3)
C19	0.245(1)	0.1033(12)	-0.318(2)	0.021(1)	0.0138(13)	0.073(4)	0.007(2)	0.055(3)	0.020(3)
C20	0.238(1)	0.0195(10)	-0.322(2)	0.022(2)	0.0066(8)	0.076(5)	0.009(2)	0.038(4)	0.004(3)
C21	0.231(1)	0.4794(9)	0.303(2)	0.016(1)	0.0098(8)	0.046(3)	0.008(2)	0.015(3)	0.018(2)
C22	0.112(1)	0.4753(9)	0.245(2)	0.021(2)	0.0062(8)	0.054(4)	0.007(2)	0.015(4)	0.005(3)
C23	0.054(1)	0.4007(10)	0.110(2)	0.017(2)	0.0078(8)	0.072(5)	0.011(2)	0.020(4)	0.015(3)
C24	0.063(1)	0.3139(9)	0.092(2)	0.019(1)	0.0068(7)	0.053(3)	0.010(2)	0.026(3)	0.015(2)
C25	0.005(1)	0.2465(10)	-0.068(2)	0.020(2)	0.0099(9)	0.034(3)	-0.003(2)	0.021(3)	0.008(2)
C26	0.040(2)	0.1549(8)	-0.073(2)	0.036(2)	0.0050(6)	0.035(3)	-0.015(2)	0.023(4)	0.001(2)
C27	0.014(1)	0.0829(12)	-0.213(2)	0.015(1)	0.0159(12)	0.069(4)	0.001(2)	0.042(3)	0.031(3)
C28	0.018(1)	0.0009(10)	-0.216(2)	0.027(2)	0.0145(9)	0.050(3)	0.027(2)	0.042(3)	0.030(3)
C29	0.482(1)	0.4947(9)	0.258(2)	0.020(1)	0.0125(9)	0.043(3)	0.016(2)	0.028(3)	0.023(2)
C30	0.506(2)	0.4070(13)	0.214(2)	0.037(2)	0.0202(12)	0.035(4)	0.042(2)	-0.007(5)	-0.014(4)
C31	0.472(2)	0.3397(10)	0.235(2)	0.050(3)	0.0077(8)	0.052(3)	-0.021(2)	0.050(4)	0.002(3)
C32	0.493(1)	0.2515(10)	0.180(2)	0.018(2)	0.0089(9)	0.037(3)	-0.003(2)	0.020(3)	0.004(3)
C33	0.440(1)	0.1855(9)	0.221(2)	0.018(2)	0.0063(7)	0.055(4)	0.007(2)	0.023(4)	0.009(3)
C34	0.451(1)	0.0937(9)	0.155(3)	0.017(2)	0.0044(7)	0.092(6)	0.002(2)	0.022(5)	0.009(3)
C35	0.393(1)	0.0200(10)	0.154(2)	0.014(1)	0.0079(9)	0.072(5)	0.006(2)	0.023(4)	0.006(3)
C36	0.269(1)	0.0201(9)	0.093(2)	0.020(2)	0.0054(7)	0.053(3)	0.001(2)	0.030(3)	0.000(3)

Table of Positional and Thermal Parameters and Their Estimated Standard Deviations (continued).

Atom	x	y	z	B, A ²	Atom	x	y	z	B, A ²
H1	0.2510(8)	0.6822(8)	0.3981(8)	5.0000(8)	H30	0.0901(8)	0.4710(8)	0.3325(8)	5.0000(8)
H2	0.2693(8)	0.8319(8)	0.4711(8)	5.0000(8)	H31	0.0701(8)	0.4194(8)	0.0220(8)	5.0000(8)
H3	0.3773(8)	0.9114(8)	0.4806(8)	5.0000(8)	H32	-0.0182(8)	0.4138(8)	0.0933(8)	5.0000(8)
H4	0.4536(8)	0.8363(8)	0.2248(8)	5.0000(8)	H33	0.0390(8)	0.3003(8)	0.1785(8)	5.0000(8)
H5	0.4482(8)	0.1419(8)	0.1419(8)	5.0000(8)	H34	0.1363(8)	0.3088(8)	0.1187(8)	5.0000(8)
H6	0.0579(8)	-0.1851(8)	-0.4823(8)	5.0000(8)	H35	0.0181(8)	0.2638(8)	-0.1518(8)	5.0000(8)
H7	0.0471(8)	-0.3347(8)	-0.5696(8)	5.0000(8)	H36	-0.0689(8)	0.2418(8)	-0.0867(8)	5.0000(8)
H8	0.1232(8)	-0.4079(8)	-0.3903(8)	5.0000(8)	H37	0.0325(8)	0.1416(8)	0.0181(8)	5.0000(8)
H9	0.2247(8)	-0.3312(8)	-0.1263(8)	5.0000(8)	H38	0.1129(8)	0.1684(8)	-0.0687(8)	5.0000(8)
H10	0.2570(8)	-0.1813(8)	-0.0371(8)	5.0000(8)	H39	-0.0059(8)	0.0959(8)	-0.3049(8)	5.0000(8)
H11	0.2820(8)	0.5195(8)	-0.1128(8)	5.0000(8)	H40	-0.0868(8)	0.0774(8)	-0.2266(8)	5.0000(8)
H12	0.1813(8)	0.4749(8)	-0.1030(8)	5.0000(8)	H41	-0.0212(8)	-0.0372(8)	-0.3235(8)	5.0000(8)
H13	0.2336(8)	0.3610(8)	-0.1392(8)	5.0000(8)	H42	-0.0040(8)	-0.0158(8)	-0.1384(8)	5.0000(8)
H14	0.3392(8)	0.4837(8)	-0.1377(8)	5.0000(8)	H43	0.5207(8)	0.5273(8)	0.2175(8)	5.0000(8)
H15	0.2742(8)	0.3935(8)	-0.3894(8)	5.0000(8)	H44	0.5094(8)	0.5157(8)	0.3751(8)	5.0000(8)
H16	0.1581(8)	0.3887(8)	-0.3945(8)	5.0000(8)	H45	0.5813(8)	0.4136(8)	0.2671(8)	5.0000(8)
H17	0.1731(8)	0.2501(8)	-0.5390(8)	5.0000(8)	H46	0.4888(8)	0.3912(8)	0.0982(8)	5.0000(8)
H18	0.1637(8)	0.2497(8)	-0.3782(8)	5.0000(8)	H47	0.3965(8)	0.3349(8)	0.1889(8)	5.0000(8)
H19	0.3370(8)	0.2468(8)	-0.2894(8)	5.0000(8)	H48	0.4936(8)	0.3536(8)	0.3515(8)	5.0000(8)
H20	0.3312(8)	0.2385(8)	-0.4663(8)	5.0000(8)	H49	0.5678(8)	0.2525(8)	0.2279(8)	5.0000(8)
H21	0.3440(8)	0.1095(8)	-0.4444(8)	5.0000(8)	H50	0.4721(8)	0.2344(8)	0.0639(8)	5.0000(8)
H22	0.2255(8)	0.1047(8)	-0.5508(8)	5.0000(8)	H51	0.3666(8)	0.1894(8)	0.1832(8)	5.0000(8)
H23	0.1769(8)	0.1196(8)	-0.3291(8)	5.0000(8)	H52	0.4673(8)	0.2001(8)	0.3374(8)	5.0000(8)
H24	0.2950(8)	0.1361(8)	-0.2124(8)	5.0000(8)	H53	0.5218(8)	0.0887(8)	0.2139(8)	5.0000(8)
H25	0.1941(8)	-0.0135(8)	-0.4318(8)	5.0000(8)	H54	0.4413(8)	0.0848(8)	0.0429(8)	5.0000(8)
H26	0.3080(8)	0.0054(8)	-0.3004(8)	5.0000(8)	H55	0.4158(8)	0.0283(8)	0.2648(8)	5.0000(8)
H27	0.2579(8)	0.5160(8)	0.4140(8)	5.0000(8)	H56	0.4087(8)	-0.0218(8)	0.0880(8)	5.0000(8)
H28	0.2440(8)	0.4217(8)	0.2979(8)	5.0000(8)	H57	0.2524(8)	0.0763(8)	0.1347(8)	5.0000(8)
H29	0.0953(8)	0.5282(8)	0.2233(8)	5.0000(8)	H58	0.2455(8)	-0.0190(8)	0.1392(8)	5.0000(8)

The form of the anisotropic thermal parameter is:
 $\exp[-(A(1,1)h^2 + A(2,2)k^2 + A(3,3)l^2 + A(1,2)hk + A(1,3)hl + A(2,3)kl)]$.

Estimated standard deviations in the least significant digits are shown in parentheses

Table of Bond Distances in Angstroms

Atom1	Atom2	Distance	Atom1	Atom2	Distance	Atom1	Atom2	Distance
SN1	C1	2.152(11)	C7	C8	1.419(15)	C22	C23	1.35(2)
SN1	C13	2.11(2)	C7	C12	1.363(14)	C23	C24	1.508(15)
SN1	C21	2.141(14)	C8	C9	1.37(2)	C24	C25	1.475(15)
SN1	C29	2.161(13)	C9	C10	1.33(2)	C25	C26	1.57(2)
SN2	C7	2.115(10)	C10	C11	1.32(2)	C26	C27	1.41(2)
SN2	C20	2.13(2)	C11	C12	1.375(14)	C27	C28	1.41(2)
SN2	C28	2.133(15)	C13	C14	1.27(2)	C29	C30	1.44(2)
SN2	C36	2.135(14)	C14	C15	1.49(2)	C30	C31	1.26(3)
C1	C2	1.344(14)	C15	C16	1.51(2)	C31	C32	1.45(2)
C1	C6	1.400(15)	C16	C17	1.39(2)	C32	C33	1.46(2)
C2	C3	1.386(15)	C17	C18	1.46(2)	C33	C34	1.467(15)
C3	C4	1.31(2)	C18	C19	1.53(2)	C34	C35	1.28(2)
C4	C5	1.34(2)	C19	C20	1.34(2)	C35	C36	1.56(2)
C5	C6	1.39(2)	C21	C22	1.50(2)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

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