AN X-RAY STRUCTURAL STUDY OF NONVALENCE INTERACTIONS AND COORDINATION IN ORGANOMETALLIC COMPOUNDS. XVII.*

THE CRYSTAL STRUCTURES OF TRIPHENYLTIN AND TRIPHENYL-LEAD QUINOLINE-8-THIOLATES

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Diffractometric data have been used to determine the crystal structures of triphenyltin quinoline-8-thiolate (I) and triphenyl-lead quinoline-8-thiolate (II) ($R_I = 0.051$ for 3748 reflections, $R_{II} = 0.062$ for 5273 reflections). In both structures the metal atoms have trigonal-bipyramidal coordination with the N atom in the axial position (Sn-N 2.602, Pb-N 2.82 Å) and the S atom in the equatorial position (Sn-S 2.434, Pb-S 2.516 Å). The slight differences in the geometric parameters of compounds I and II indicates that the coordination interaction in compound I is slightly stronger than that in compound II, as confirmed by the exchange equilibria.

Up to the present, there have been no published structural studies indicating the possibility of formation of chelate rings in trialkyltin and triaryltin derivatives of o-substituted thiophenols and their organolead analogs. From the ^{19}F NMR spectra, indications have been obtained of the existence in solution of intramolecular coordination in $(C_6H_5)_3Pb$ and $(C_6H_5)_3Sn$ derivatives of 2,6-dibromo-4-fluorothiophenol [2]. An x-ray structural analysis of the last compound showed, however, that there is no intramolecular coordination in the crystal [3]. The study of exchange equilibria involving organometallic derivatives of o-substituted thiophenols has established that in most cases the ability to undergo intramolecular coordination increases with change in the nature of the organometallic group in the series $(C_6H_5)_3Sn < (C_6H_5)_3Pb < C_6H_5Hg$ [4] and that in the $(C_6H_5)_3Sn$ derivative of o-chlorothiophenol, there is practically no intramolecular coordination [5]. At the same time, the Mössbauer spectra (6) and an x-ray structural analysis [7] indicate that in compounds containing a tin–sulfur bond, the $(C_6H_5)_3Sn$ group is capable of intensive intermolecular coordination with the nitrogen atom of the pyridine ring in the crystalline state. According to the Mössbauer spectra [6] and the ^{19}F NMR spectra [8], coordination interactions of this kind can also take place in solution.

In this connection, it was of interest to investigate the possibility of formation of chelate rings containing the $(C_6H_5)_3Sn$ group and a pyridine nitrogen atom in compounds with a tin–sulfur bond, and also to com-

TABLE 1. Crystallograhic Data for Compounds I and II

Parameter	I	II	Parameter	I	II
a, Å	9,672(8)	9,720(1)	V, Å³	2282,0	2309,8
b, Å	10,252(8)	10,297(1)	d_{calc}, g/cm³	1,485	1,722
c, Å	25,38(2)	25,370(4)	Z	4	4
α, deg	90,44(7)	90,41(1)	Space group	$P\bar{1}$	$P\bar{1}$
β, deg	93,29(7)	93,90(1)	$\mu(\lambda Mo)$, cm⁻¹	12,3	73,0
γ, deg	114,67(6)	114,16(1)			

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*For Part XVI see [1].

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TABLE 2. Atom Coordinates in the Structure of Compound I ($\times 10^4$)

Molecule A				Molecule B			
atom	x	y	z	atom	x	y	z
Sn	2856,5(8)	6369,4(7)	3745,4(3)	Sn′	−1762,3(7)	1223,8(7)	1308,8(3)
S	4319(3)	8039(3)	4472(1)	S′	−3373(3)	1045(3)	517(1)
N	5667(9)	7433(9)	3495(3)	N′	−759(9)	3787(8)	924(3)
C(1)	6224(11)	8855(10)	4295(4)	C(1′)	−2972(12)	2817(11)	331(4)
C(2)	7293(12)	9895(11)	4658(5)	C(2′)	−3984(13)	3018(13)	−46(5)
C(3)	8857(13)	10561(12)	4522(6)	C(3′)	−3669(15)	4399(14)	−228(5)
C(4)	9343(12)	10223(11)	4062(5)	C(4′)	−2411(14)	5552(13)	−41(5)
C(5)	8271(11)	9149(10)	3706(5)	C(5′)	−1371(12)	5392(11)	342(4)
C(6)	8694(13)	8735(12)	3236(5)	C(6′)	−10(13)	6549(12)	553(5)
C(7)	7628(13)	7690(12)	2908(5)	C(7′)	933(13)	6308(11)	915(5)
C(8)	6088(12)	7053(12)	3063(5)	C(8′)	505(12)	4917(12)	1106(5)
C(9)	6705(11)	8465(10)	3824(4)	C(9′)	−1675(11)	4005(10)	547(4)
C(10)	692(11)	5776(10)	4087(4)	C(10′)	−3009(11)	−1062(10)	1435(5)
C(11)	−165(12)	4357(11)	4220(5)	C(11′)	−4454(12)	−1525(11)	1643(5)
C(12)	−1581(13)	3980(13)	4440(6)	C(12′)	−5298(13)	−3005(12)	1727(5)
C(13)	−2133(12)	5010(13)	4535(5)	C(13′)	−4729(15)	−3990(12)	1598(5)
C(14)	−1271(13)	6430(13)	4411(5)	C(14′)	−3308(14)	−3535(12)	1363(6)
C(15)	128(12)	6798(12)	4183(5)	C(15′)	−2433(13)	−2072(11)	1310(5)
C(16)	2466(11)	7291(10)	3029(4)	C(16′)	498(11)	1434(10)	1221(4)
C(17)	3496(12)	8685(11)	2870(5)	C(17′)	1624(12)	2077(11)	1636(5)
C(18)	3107(14)	9288(12)	2445(5)	C(18′)	3091(13)	2116(12)	1591(6)
C(19)	1738(14)	8558(13)	2135(5)	C(19′)	3419(13)	1560(13)	1158(7)
C(20)	748(13)	7170(13)	2277(5)	C(20′)	2340(16)	929(14)	724(6)
C(21)	1101(12)	6566(12)	2730(5)	C(21′)	822(13)	866(12)	761(5)
C(22)	3117(12)	4395(11)	3681(5)	C(22′)	−2126(10)	2209(10)	2004(4)
C(23)	3720(13)	3959(12)	4129(5)	C(23′)	−1931(15)	1667(13)	2485(5)
C(24)	3868(14)	2644(13)	4100(6)	C(24′)	−2210(16)	2238(14)	2963(5)
C(25)	3409(13)	1817(13)	3618(6)	C(25′)	−2760(14)	3286(13)	2946(5)
C(26)	2748(16)	2233(14)	3193(6)	C(26′)	−3005(12)	3787(12)	2447(5)
C(27)	2626(15)	3542(12)	3220(5)	C(27′)	−2680(11)	3272(11)	1983(4)

pare the structures of these compounds with the structures of their organolead analogs. For this purpose, an x-ray structural analysis of triphenyltin quinoline-8-thiolate (I) and triphenyl-lead quinoline-8-thiolate (II) was carried out.

EXPERIMENTAL

Compounds I and II were obtained by the reaction of the corresponding organometallic chlorides with sodium quinoline-8-thiolate in alcoholic solution, and purified by recrystallization.

Compound I forms yellow crystals with mp 170-172° after crystallization from propanol. Found: C 63.34, H 4.01, S 6.65, Sn 23.38%. Calculated for $C_{27}H_{21}NSSn$: C 63.53, H 4.10, S 6.27, Sn 23.33%.

Compound II forms yellow crystals with mp 110-112° after crystallization from methanol. Found: C 54.12, H 3.58, S 5.33, Pb 34.76%. Calculated for $C_{27}H_{21}NPbS$: C 54.18, H 3.68, S 5.36, Pb 34.61%.

Compounds I and II are isostructural; the crystals are triclinic, and the crystallographic data are given in Table 1.

The intensities of the reflections were measured on a Syntex $P2_1$ automatic four-circle diffractometer using Mo radiation and a graphite monochromator by the $\theta/2\theta$ scanning method in the range $2° \le 2\theta \le 42°$ for compound I and $2° \le 2\theta \le 55°$ for compound II. A total of 3748 reflections for compound I and 5273 reflections for compound II with $F^2 > 2\sigma$ was obtained. For compound II, the method of Gaussian integration using the modified program ORABS [9] was used to take account of absorption for the real form of the crystal, described by nine faces.

The structure of compound I was determined by the heavy atom method and refined by the method of least squares first in the full-matrix isotropic approximation and then in the block-diagonal anisotropic approximation with weighting scheme $1/w = (\sigma F)^2 + (0.025F_{meas})^2$; the final value of R was 0.051.

For structure II, the coordinates of the atoms of compound I were taken as the starting coordinates. Refinement by an analogous scheme gave R = 0.062. For both structures, corrections were introduced for the

TABLE 3. Atom Coordinates in the Structure of Compound II ($\times 10^4$)

Molecule A				Molecule B			
atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
Pb	2678,3(5)	6235,3(5)	3745,5(2)	Pb′	—1727,7(4)	1101,8(5)	1350,4(2)
S	4325(4)	8019(4)	4459(2)	S′	3286(3)	1062(4)	515(2)
N	5707(10)	7363(11)	3493(4)	N′	—629(9)	3841(9)	909(4)
C(1)	6186(12)	8835(12)	4271(5)	C(1′)	—2915(12)	2817(13)	347(5)
C(2)	7237(13)	9888(13)	4629(6)	C(2′)	—3958(14)	3031(16)	—22(5)
C(3)	8768(14)	10586(14)	4510(6)	C(3′)	—3703(18)	4385(18)	—200(6)
C(4)	9258(14)	10198(12)	4064(6)	C(4′)	—2406(16)	5576(15)	—20(6)
C(5)	8251(12)	9129(12)	3706(5)	C(5′)	—1368(14)	5376(14)	343(5)
C(6)	8698(14)	8702(15)	3243(6)	C(6′)	27(14)	6578(14)	534(6)
C(7)	7670(14)	7626(14)	2909(6)	C(7′)	1002(14)	6351(13)	8941(6)
C(8)	6161(14)	6975(14)	3062(6)	C(8′)	639(13)	4966(13)	1083(6)
C(9)	6714(12)	8451(12)	3818(5)	C(9′)	—1614(12)	4034(12)	547(4)
C(10)	514(11)	5712(11)	4124(4)	C(10′)	—3041(11)	—1242(11)	1458(5)
C(11)	—322(13)	4310(13)	4231(5)	C(11′)	—4467(13)	—1661(13)	1664(6)
C(12)	—1730(14)	3932(15)	4445(6)	C(12′)	—5350(14)	—3073(14)	1741(6)
C(13)	—2240(14)	5032(16)	4535(6)	C(13′)	—4831(16)	—4096(14)	1599(7)
C(14)	—1377(14)	6403(15)	4433(6)	C(14′)	—3415(17)	—3683(14)	1375(7)
C(15)	—8(13)	6741(15)	4212(6)	C(15′)	—2530(13)	—2247(12)	1306(5)
C(16)	2371(11)	7231(11)	3003(5)	C(16′)	609(11)	1384(11)	1224(5)
C(17)	3415(12)	8568(12)	2878(5)	C(17′)	1719(12)	2011(12)	1648(6)
C(18)	3100(16)	9238(14)	2437(6)	C(18′)	3173(14)	2078(14)	1589(8)
C(19)	1728(15)	8523(15)	2100(6)	C(19′)	3462(14)	1522(16)	1129(8)
C(20)	740(14)	7147(15)	2241(6)	C(20′)	2390(17)	911(17)	742(8)
C(21)	1038(13)	6523(15)	2694(5)	C(21′)	841(15)	790(15)	767(6)
C(22)	3068(12)	4296(12)	3704(5)	C(22′)	—2139(11)	2167(11)	2044(5)
C(23)	3588(14)	3812(15)	4151(6)	C(23′)	—2053(16)	1615(14)	2537(6)
C(24)	3795(16)	2513(16)	4128(7)	C(24′)	—2400(19)	2198(17)	2981(6)
C(25)	3418(15)	1718(15)	3659(7)	C(25′)	—2820(15)	3318(14)	2937(6)
C(26)	2867(18)	2152(15)	3220(8)	C(26′)	—2944(12)	3826(13)	2443(6)
C(27)	2612(16)	3455(15)	3236(6)	C(27′)	—2630(12)	3261(12)	1992(5)

anomalous scattering by the M and S atoms.

The atom coordinates are given in Tables 2 and 3; the tables to temperature factors can be obtained from the authors.

GEOMETRY OF THE MOLECULES

The crystals of compounds I and II contain two crystallographically independent molecules (A and B). The structures of both the independent molecules in each structure and the molecules of compounds I and II are very similar, and are shown in Fig. 1. The slight difference in their conformations can be characterized by the torsional angles at the M—S bonds (Table 4). The principal bond lengths and valence angles are given in Table 5. In the phenyl rings, the average value of the bond lengths in compounds I and II is 1.40 Å (scatter 1.35-1.45 Å in compound I and 1.35-1.46 Å in compound II), and the average value of the valence angles is 120° (118-123° and 114-123° in compounds I and II respectively).

The metal atom in both structures forms a covalent bond with the S atom and a strong coordinate bond with the N atom, and has trigonal-bipyramidal coordination. The rigidity of the chelate quinolinethiolate ligand makes it impossible to achieve the arrangement of the more electronegative substituents N and S in the axial positions, optimal for the trigonal-bipyramidal complexes, and these positions are occupied by the N and C(10) atoms, and the atoms S, C (16), and C(22) are in the equatorial positions. Similar coordination was previously found in triphenyltin N-benzoyl-N-phenylhydroxylaminate [10]. All three structures show a slight increase (by 0.03-0.04 Å) in the length of the axial M—C bond compared with the equatorial bonds.

The bond lengths Sn—S (average 2.434 Å) and Pb-S (average 2.516 Å) are close to the corresponding values in the CH_3-substituted thiophenolates of triphenyltin and triphenyllead (Sn—S 2.434(5) [11] and 2.420(8) Å [3], Pb-S 2.51(6) Å [12]), but much shorter than those in the triphenyltin (III) and triphenyllead (IV) derivatives of 4-thiopyridone (Sn-S, 2576(7) Å [7], Pb-S 2.68(1] Å [13]). The distances Sn...$\overline{N}$ (average 2.602 Å) and Pb—N (average 2.82 Å) practically coincide with Sn...N 2.62(2) Å in compound III and Pb...N 2.76(3) Å in compound IV. Thus the coordination interactions in compounds I and II on the one hand and in compounds

TABLE 4. Torsional Angles φ° at the M−S Bonds in the Molecules of Compounds I and II

Atom	φ			
	I A	I B	II A	II B
C(1)—S—M—C(10)	−176,1	169,3	−174,6	167,3
C(1)—S—M—C(16)	−71,9	−85,6	−67,1	−83,9
C(1)—S—M—C(22)	75,2	64,0	73,2	59,4

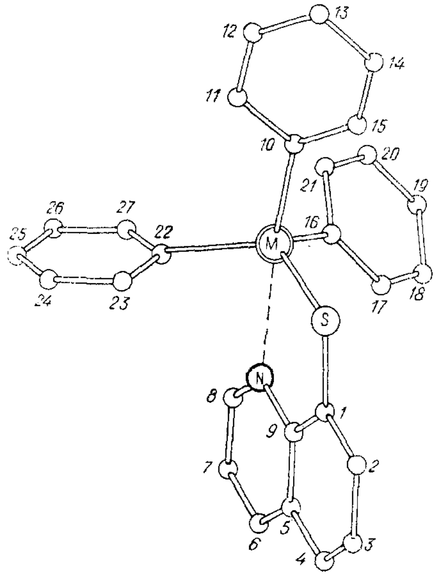

Fig. 1. Structure of molecules I and II and the numbering of the atoms.

III and IV on the other are of approximately equal strength, but it is only in the potentially metallogropic structures of compounds III and IV, where rearrangement of the π-electron system is possible, that this interaction leads to a sharp increase in the length of M−S bonds, and in compounds I and II, these bonds, like the S−C bonds, remain normal single covalent bonds.

Although the structures of the molecules of compounds I and II are qualitatively identical, the structures show quantitative differences, indicating a slightly stronger coordination interaction in compound I compared with compound II. First of all, in compound I the distance Sn...N is greater than the sum of the covalent radii by 0.50 Å, whereas in compound II, Pb...N is slightly greater, by 0.66 Å; correspondingly, compared with the sum of the van der Waals radii,* the distance M...N is shortened by ~1.2 Å in compound I, and by ~0.8 Å in compound II, that is this bond is closer to covalent in compound I than in compound II. Moreover, in compound I the trigonal bipyramid at the metal atom is less distorted than in compound II: The angles between the axial bonds are closer to linear (average values 166.6 in compound I and 163.8° in compound II), and the sum of the angles between the equatorial bonds is closer to 360° (352.0 and 347.4° respectively).

Averaging of the bond lengths in the quinolinethiolate ring over the four molecules makes it possible to state that they are quite definitely unequal, and the distribution of the longer and shorter bonds is similar in character to that in 8-quinolinol [15] and the quinolinolato-complexes of metals [16], whereas in the quinolinethiolato-complexes $Hg(C_9H_6NS)_2$, $Pb(C_9H_6NS)_2$ and $Sb(C_9H_6NS)_3$ [17], the bonds in the ring vary in a narrower range (Table 6).

The quinolinethiolate rings in compounds I and II are approximately planar; the deviations of the atoms from the average plane of the bicyclic system do not exceed 0.04 Å. The S atoms in the molecules A in both compound I and compound II are situated in this plane (deviations 0.001(3) Å in compound I and −0.012(4) Å in compound II), and the M atoms are only slightly displaced from it: Sn by 0.1089(7) Å, and Pb by 0.1521(5)

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*The following values of the van der Waals radii were used: Sn 2.2 Å [3], Pb 2.0 Å [12], N 1.6 Å [14].

TABLE 5. Principal Bond Lengths d, Å and Valence Angles ω, deg, in the Structures of Compounds I and II

Atom	d				Atom	d			
	I A	I B	II A	II B		I A	I B	II A	II B
M—S	2,441(3)	2,427(3)	2,522(4)	2,509(4)	C(1)—C(2)	1,42(2)	1,41(2)	1,41(2)	1,42(2)
M—N	2,592(9)	2,611(8)	2,81(1)	2,84(1)	C(2)—C(3)	1,44(2)	1,41(2)	1,42(2)	1,40(2)
M—C(10)	2,16(1)	2,18(1)	2,24(1)	2,25(1)	C(3)—C(4)	1,38(2)	1,35(2)	1,38(2)	1,40(2)
M—C(16)	2,14(1)	2,13(1)	2,21(1)	2,22(1)	C(4)—C(5)	1,42(2)	1,42(2)	1,41(2)	1,40(2)
M—C(22)	2,15(1)	2,15(1)	2,18(1)	2,21(1)	C(5)—C(6)	1,40(2)	1,43(2)	1,41(2)	1,46(2)
S—C(1)	1,76(1)	1,77(1)	1,76(1)	1,75(1)	C(6)—C(7)	1,37(2)	1,35(2)	1,38(2)	1,36(2)
N—C(8)	1,30(2)	1,34(2)	1,32(2)	1,34(2)	C(7)—C(8)	1,43(2)	1,41(2)	1,42(2)	1,42(2)
N—C(9)	1,35(2)	1,35(2)	1,37(2)	1,36(2)	C(1)—C(9)	1,42(2)	1,41(2)	1,41(2)	1,42(2)
					C(9)—C(5)	1,43(2)	1,43(2)	1,42(2)	1,41(2)

Atom	ω				Atom	ω			
	I A	I B	II A	II B		I A	I B	II A	II B
SMN	74,4(2)	73,4(2)	70,1(2)	69,6(2)	C(9)C(1)C(2)	120,5(1,0)	120,3(1,0)	118,6(1,1)	118,1(1,2)
SMC(10)	94,4(3)	91,1(3)	95,4(3)	92,6(3)	C(1)C(2)C(3)	117,6(1,1)	120,0(1,2)	119,6(1,2)	121,3(1,3)
SMC(16)	116,8(3)	118,3(3)	113,2(3)	113,9(3)	C(2)C(3)C(4)	122,9(1,2)	121,0(1,3)	120,7(1,3)	120,1(1,5)
SMC(22)	116,9(3)	117,7(3)	114,0(3)	115,1(3)	C(3)C(4)C(5)	119,2(1,1)	120,5(1,2)	121,1(1,3)	118,1(1,4)
NMC(10)	168,7(3)	164,4(4)	165,5(4)	162,1(4)	C(4)C(5)C(6)	122,2(1,0)	123,9(1,1)	123,1(1,2)	120,5(1,3)
NMC(16)	84,3(3)	84,2(3)	79,5(4)	80,3(4)	C(4)C(5)C(9)	119,7(1,0)	120,0(1,0)	118,4(1,1)	122,5(1,2)
NMC(22)	81,8(4)	85,7(3)	82,5(4)	85,5(4)	C(9)C(5)C(6)	118,1(1,0)	116,0(1,0)	118,5(1,2)	117,0(1,2)
C(10)MC(16)	100,5(4)	102,8(4)	103,8(4)	106,0(4)	C(5)C(6)C(7)	120,2(1,1)	120,5(1,1)	120,9(1,3)	119,0(1,3)
C(10)MC(22)	104,5(4)	103,0(4)	107,7(4)	105,0(4)	C(6)C(7)C(8)	117,7(1,1)	119,1(1,2)	116,6(1,3)	119,8(1,3)
C(16)MC(22)	117,6(4)	116,8(4)	119,0(4)	119,5(4)	C(7)C(8)N	123,2(1,1)	123,1(1,1)	123,7(1,3)	122,3(1,2)
MSC(1)	106,7(4)	107,0(4)	109,7(4)	109,2(4)	C(8)NC(9)	120,0(9)	118,1(9)	119,8(1,1)	119,2(1,0)
MNC(8)	122,9(7)	125,1(7)	123,5(8)	125,6(8)	NC(9)C(5)	120,8(8)	123,0(9)	120,4(1,1)	122,7(1,1)
MNC(9)	117,0(6)	116,4(6)	116,5(8)	114,5(7)	NC(9)C(1)	119,2(9)	119,0(9)	118,0(1,1)	118,4(1,0)
SC(1)C(2)	116,9(8)	118,0(9)	116,1(9)	117,7(1,0)	C(5)C(9)C(1)	120,0(9)	117,9(1,0)	121,5(1,1)	118,8(1,1)
SC(1)C(9)	122,5(8)	121,7(8)	125,2(9)	124,2(9)					

TABLE 6. Bond Lengths d, Å in Quinolinethiolate and Quinolinolate Rings

Atom	d				Atom	d			
	a	b	c	d		a	b	c	d
C(1)—C(2)	1,41	1,380	1,39	1,40	C(7)—C(8)	1,42	1,411	1,40	1,42
C(2)—C(3)	1,42	1,429	1,42	1,42	C(9)—C(1)	1,42	1,411	1,41	1,41
C(3)—C(4)	1,38	1,364	1,37	1,41	C(9)—C(5)	1,42	1,431	1,42	1,42
C(4)—C(5)	1,41	1,418	1,40	1,40	N—C(8)	1,32	1,318	1,33	1,34
C(5)—C(6)	1,42	1,426	1,42	1,42	N—C(9)	1,36	1,372	1,37	1,37
C(6)—C(7)	1,36	1,352	1,38	1,39					

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a) Average values in the structures of compounds I and II; b) 8-quinolinol [15]; c) average values in quinolinolato-complexes from the data in [16]; d) average values in quinolinethiolato-complexes from the data in [17].

Å. In the molecules B, the S atoms are displaced from the corresponding plane by 0.167(3) Å in compound I and 0.180(4) Å in compound II, and the metal atoms are displaced by −0.3552(7) Å in compound I and −0.4934(5) Å in compound II.

The results of the x-ray structural analysis of compounds I and II, which indicate that the intensity of the intramolecular coordination is greater in the first case, are confirmed by the exchange equilibria. Thus the equilibrium constant, determined by PMR, for the exchange reaction of compound II with the $(C_6H_5)_3Sn$ derivative of 2,6-dimethylthiophenol in chloroform is 22. According to data previously obtained [4, 8], this result confirms unambiguously that the chelate ring in compound I is more stable than that in compound II. This feature apparently reflects the greater "hardness," in the terminology of Pearson's theory [18], of the $(C_6H_5)_3Sn$ group as a Lewis acid, compared with the $(C_6H_5)_3Pb$ group. Comparison of the results obtained with the results of earlier studies [4] shows that the order of relative strength of the chelate rings formed by the $(C_6H_5)_3Sn$ and $(C_6H_5)_3Pb$ groups may be reversed when there is a change in the geometry of the donor–acceptor interactions and the nature of the donor of the unshared electron pair.

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