^{13}C AND ^{119}Sn NMR SPECTRA OF SOME MONO-n-BUTYLTIN(IV) COMPOUNDS

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The ^{13}C and ^{119}Sn NMR spectra of some mono-n-butyltin(IV) compounds in solution of non-coordinating ($CDCl_3$ and CD_3NO_2) and coordinating ($(CD_3)_2SO$ and C_5D_5N) solvents have been studied. From the values of $\delta(^{119}Sn)$, $^1J(^{119}Sn,^{13}C)$ and additional ^{13}C NMR parameters an evaluation of the coordination numbers of the central tin atom and the shapes of coordination polyhedra around the tin atom has been carried out.

In the series of our previous papers[1–7] we have analyzed some parameters of ^{13}C and ^{119}Sn NMR spectra of organotin(IV) compounds of the type R_nSnX_{4-n} and their complexes $R_nSnX_{4-n} . x\,D$, where R is n-butyl, vinyl, phenyl and benzyl, X is a polar group and D is an equivalent of the unidentate Lewis base, $n = 2$ or 3, $x = 1–3$, in order to find relations between their structures and these NMR spectra parameters. From this analysis we have concluded that for a qualitative description of the geometry of coordination polyhedra around Sn atom (or at least a skeleton of R_nSn), it is sufficient to use two parameters – the chemical shift $\delta(^{119}Sn)$ and the coupling constant $^1J(^{119}Sn,^{13}C)$. Moreover, the value of $\delta(^{119}Sn)$ is directly linked to the coordination number of the central tin atom, the value of $^1J(^{119}Sn,^{13}C)$ depends linearly on the value of bonding angles C–Sn–C (refs[8–11]).

There is a question now, if analogous parameters of ^{119}Sn and ^{13}C NMR spectra are also suitable for the prediction of the structure of monoorganotin compounds and their complexes. A partial answer could be obtained from the study of a set of mono-n-butyltin(IV) compounds presented in this paper.

EXPERIMENTAL

Compounds *I–III*, *V*, *X*, *XII*, *XIV–XVI*, and *XVIII* (see Tables I and II) were prepared according to published procedures[12–19]. Compound *IV* was synthesized by reacting tetraethylammonium chloride (4.0 g, 21.2 mmol) with mono-n-butyltin(IV) trichloride (3.0 g, 10.6 mmol) in 25 ml of 2-propanol.

After stirring the reaction mixture for 1 h and cooling down to 5 °C, small white crystals of the product were obtained and recrystallized from the same solvent. Compound *VI* was obtained by reaction of sodium 1,2-ethanedithiolate (2.9 g, 21 mmol) with mono-n-butyltin(IV) trichloride (6 g, 21 mmol) in 50 ml of methanol. After stirring the mixture for 1 h at the room temperature, the solvent was distilled off in vacuo. The white suspension obtained was extracted with chloroform and after the distillation off the solvent from the extract, a white oily liquid was obtained and redistilled in vacuo. Compound *VII* was prepared likewise from mono-n-butyltin(IV) tribromide. Compounds *VIII* and *IX* were prepared by the procedure described for *X* (ref.[16]) by reacting mono-n-butyltin(IV) trichloride with silver acetate in the molar ratio of 1 : 1 and 1 : 2, respectively. Analogously to the procedure described for *XII* (ref.[17]), *XIII* was prepared from mono-n-butyltin(IV) tribromide and *XI* from mono-

TABLE I
Analytical and physical data for compounds *IV, VI–IX, XI, XIII, XVII and XIX*

Compound[a]	Formula M.w.	M.p. (b.p.) °C/Pa	Calculated/Found					
			% C	% H	% Sn	% others		
IV	$C_{20}H_{49}Cl_5N_2Sn$	199–201	39.15	8.05	19.34	N: 4.57	Cl: 28.89	
	613.6		39.10	8.52	18.81	3.70	28.94	
VI	$C_6H_{13}ClS_2Sn$	(140–145/400)	23.75	4.32	39.12	S: 21.13	Cl: 11.68	
	303.4		23.82	4.35	38.94	21.25	11.36	
VII	$C_6H_{13}BrS_2Sn$	(136–139/250)	20.72	3.77	34.12	S: 18.43	Br: 22.97	
	347.9		20.97	3.85	33.85	18.57	22.63	
VIII	$C_6H_{12}Cl_2O_2Sn$	[b]	23.57	3.96	38.82	Cl: 23.19		
	305.8		23.20	3.89	39.06	22.89		
IX	$C_8H_{15}ClO_4Sn$	[b]	29.18	4.59	36.04	Cl: 10.76		
	329.3		29.51	4.64	35.91	10.71		
XI	$C_9H_{19}Cl_2NS_2Sn$	53–56	27.37	4.85	30.05	Cl: 17.95	N: 3.55	S: 16.23
	395.0		27.51	4.82	29.87	17.84	3.71	16.05
XIII	$C_9H_{13}Br_2NS_2Sn$	67–70	22.34	3.96	24.53	Br: 33.03	N: 2.89	S: 13.25
	483.9		22.27	4.01	24.82	32.89	3.01	13.10
XVII	$C_{22}H_{21}BrN_2O_2S$	171–173	48.57	3.89	21.82	Br: 14.69	N: 5.15	
	544.0		48.47	4.13	21.49	14.75	5.16	
XIX	$C_{22}H_{21}ClN_2S_2Sn$	164–166	49.70	3.98	22.32	Cl: 6.67	N: 5.27	S: 12.06
	531.7		49.43	4.60	21.20	7.05	5.82	11.99

[a] See Table II. [b] Yellow liquid, not purified due to its very low thermal stability.

TABLE II
^{13}C and ^{119}Sn NMR parameters of compounds *I*–*XIX* at 300 K

Comp.[a]	Formula	$\delta(^{119}Sn)$ ppm	$\delta(^{13}C)$, ppm/$^nJ(^{119}Sn,^{13}C)$, Hz				Others
			$n = 1$	$n = 2$	$n = 3$	$n = 4$	
Ia	$BuSnCl_3$[b]	6.1	33.70	26.71	25.40	13.10	
			(645.0)	(40.0)	(120.0)	[d]	
Ib	$BuSnCl_3$[c]	–457.1	44.75	28.28	25.38	14.19	
			[d]	(71.6)	(210.3)	(12.6)	
IIa	$BuSnBr_3$[b]	–114.7	34.76	27.40	25.12	13.37	
			(590.6)	(56.8)	(117.8)	[d]	
IIb	$BuSnBr_3$[c]	–604.1	39.64	28.02	24.09	13.52	
			[d]	(69.7)	(218.9)	[d]	
III	$BuSnI_3$[c]	–577.8	33.20	29.34	23.74	13.68	
			(455.6)	(48.8)	(118.4)	[d]	
IV	$[Et_4N]_2[BuSnCl_5]$[e]	–454.2	55.71	29.80	26.16	14.06	53.32 (1′), 7.62 (2′)
			(1 234.0)	(72.1)	(253.1)	[d]	
Va	$BuSn(SBu)_3$[b]	147.9	22.38	27.79	26.14	13.49	36.17 (1′), 27.49 (2′)
			(445.2)	(32.9)	(83.8)	[d]	21.53 (3′), 13.39 (4′)
Vb	$BuSn(SBu)_3$[f]	142.2	22.61	28.07	26.21	13.42	36.43 (1′), 27.67 (2′)
			(451.5)	(33.1)	(81.2)	[d]	21.68 (3′), 13.47 (4′)
VIa	BuSn(dtg)Cl[b]	–12.3	30.20	27.29	25.51	13.17	35.46 (1′)
			(559.0)	(44.3)	(99.5)	[d]	
VIb	BuSn(dtg)Cl[c]	–446.8	34.20	28.26	25.68	14.00	33.43 (1′)
			(807.0)	(54.1)	(131.9)	(7.3)	
VII	BuSn(dtg)Br[b]	158.9	30.20	27.71	25.61	13.31	36.03 (1′)
			(502.3)	(42.8)	(95.9)	[d]	
VIII	$BuSn(OAc)Cl_2$[b]	–161.5	33.80	26.41	25.11	13.10	183.45 (1′),
			(771.0)	(61.8)	(141.8)	(7.3)	18.90 (2′)
IX	$BuSn(OAc)_2Cl$[b]	–350.5	32.09	26.16	24.96	13.11	183.74 (1′),
			[d]	(62.4)	(172.3)	[d]	18.70 (2′)
X	$BuSn(OAc)_3$[b]	–533.1	28.07	25.72	24.69	12.79	183.44 (1′),
			(1 014.4)	(61.1)	(171.1)	(10.2)	17.90 (2′)
XI	$BuSn(edtc)Cl_2$[b]	–299.1	36.65	27.05	24.96	13.31	191.65 (1′)
			(757.7)	(57.5)	(141.4)	[d]	51.62 (2′), 11.74 (3′)
XII	$BuSn(edtc)_2Cl$[b]	–579.8	46.93	28.17	25.04	13.57	197.14 (1′)
			(945.0)	(63.2)	(213.0)	[d]	50.74 (2′), 11.86 (3′)
XIII	$BuSn(edtc)Br_2$[b]	–339.2	38.43	27.59	24.61	13.39	192.37 (1′)
			(715.1)	(59.0)	(145.8)	[d]	51.46 (2′), 11.86 (3′)
XIV	$BuSn(edtc)_2Br$[b]	–619.4	49.01	28.31	24.79	13.64	197.55 (1′)
			(925.0)	(64.7)	(215.3)	(11.2)	50.78 (2′), 11.91 (3′)

TABLE II
(*Continued*)

Comp.[a]	Formula	$\delta(^{119}Sn)$ ppm	$\delta(^{13}C)$, ppm/$^nJ(^{119}Sn,^{13}C)$, Hz				Others
			n = 1	n = 2	n = 3	n = 4	
XV	$BuSn(edtc)_3$[b]	–660.3	51.84	29.05	25.11	13.72	200.61 (1′),
			(975.8)	(73.0)	(230.6)	[d]	49.80 (2′), 12.05 (3′)
XVI	$BuSn(oxin)_2Cl$[b]	–395.6	29.14	26.97	25.56	13.11	[g]
			(1 066.6)	(54.3)	(114.2)	(10.8)	
XVII	$BuSn(oxin)_2Br$[b]	–428.3	32.02	27.53	25.75	13.48	[h]
			(1 036.0)	(54.7)	(175.4)	(11.6)	
XVIII	$BuSn(oxin)_3$[b]	–561.2	29.00	27.00	25.79	13.22	[i]
			(1 206.2)	(59.0)	(181.6)	(12.5)	
XIX	$BuSn(txin)_2Cl$[b]	–275.9	40.33	28.21	25.95	13.53	[k]
			(789.3)	(49.9)	(165.1)	(9.5)	

[a] Bu = $–CH_2^{1}–CH_2^{2}–CH_2^{3}–CH_3^{4}$, SBu = $–S–CH_2^{1'}–CH_2^{2'}–CH_2^{3'}–CH_3^{4'}$, Et = $–CH_2^{1'}–CH_3^{2'}$, dtg = $–SCH_2^{1'}–CH_2^{1'}S–$, Ac = $CH_3^{2'}–C^{1'}OO$, edtc = $(CH_3^{3'}–CH_2^{2'})_2NC^{1'}S_2$, oxin = 8-quinolinate, txin = 8-quinolinethiolate. [b] In $CDCl_3$. [c] In hexadeuteriodimethyl sulfoxide. [d] Not found. [e] In trideuterionitromethane. [f] In pentadeuteriopyridine. [g–k] $\delta(^{13}C)$: [g] 156.26, 154.66, 142.85 (2 ×), 139.94 (2 ×), 135.52, 133.86, 130.51, 130.01, 129.36, 129.03, 121.18 (2 ×), 114.18, 113.71, 113.44 (2 ×); [h] 156.77, 154.95, 143.20 (2 ×), 140.27 (2 ×), 135.95, 133.92, 130.88, 130.45, 129.69, 129.46, 121.67, 121.37, 114.66 (2 ×), 113.97 (2 ×); [i] 157.60, 145.42, 138.37, 137.16, 129.72, 129.01, 120.85, 112.87, 111.40; [k] 147.32, 141.27, 139.50, 138.36, 131.17, 129.65, 127.90, 122.78, 121.26.

n-butyltin(IV) trichloride and sodium diethyldithiocarbamate in the molar ration 1 : 1. According to the procedure described for *XV* (ref.[18]), compounds *XVII* and *XIX* were prepared from mono-n-butyltin(IV) trichloride and mono-n-butyltin(IV) tribromide, respectively, and sodium salts of 8-hydroxyquinoline and 8-quinolinethiol in the molar ratio 1 : 2.

The analytical data and some physical data of the new compounds prepared are summarized in Table I.

^{119}Sn (134.29 MHz) and ^{13}C (90.566 MHz) spectra were recorded at 300 K on a Bruker AMX 360 spectrometer equipped with 5 mm multinuclear tuneable probe and X32 computer using the UXNMR software (Version 940501.3). Compounds prepared were measured in $CDCl_3$, CD_3NO_2, C_5D_5N or $(CD_3)_2SO$ the concentrations being 5–30%. The ^{13}C chemical shifts were referred to appropriate signals of solvents and recalculated to δ-scale ($\delta(^{13}C)$ = 77.00 ($CDCl_3$); 39.60 ($(CD_3)_2SO$); 62.8 (CD_3NO_2); 149.5 (C-2 in C_5D_5N). ^{119}Sn chemical shifts were referred to external neat $Sn(CH_3)_4$ (δ = 0.0) placed in a coaxial capillary.

RESULTS AND DISCUSSION

The values of ^{13}C and ^{119}Sn NMR parameters of the compounds studied in this paper are given in Table II (compounds *I–XIX*). The number of $\delta(^{119}Sn)$ values (within the intervals of 25 ppm) of compounds measured in this paper as well as of data for other mono-n-butyltin compounds obtained from a careful inspection of literature[20–30], is plotted in Fig. 1 for the coordination numbers of 4–7. It can be seen that δ(^{119}Sn) of the compounds *I*–*XIX* cover practically the whole range of the previously observed values of the chemical shift of mono-n-butyltin compounds and the compounds of *V* and *VII* even expand this range downfield. The ^{119}Sn chemical shift in *VII* (+158.9 ppm) has been the most positive value observed up to now for mono-n-butyltin(IV) derivatives.

From Table II and also from Fig. 1 it can be seen that the values of δ(^{119}Sn) are shifted upfield with an increasing coordination number of the tin atom similarly like in the tri- and di-n-butyltin compounds and, in fact, in all organotin compounds[1–7]. But the ranges of the δ(^{119}Sn) values characteristic of appropriate coordination numbers are relatively broad and are not mutually separated one from another, contrary to the tri- and di-n-butyltin compounds and another tri- and diorganotin compounds. This fact is unambiguously associated with the concerted influence of several factors, others than only the coordination number of the tin atom. In tri- and diorganotin compounds these effects influence δ(^{119}Sn) to the relatively small extent only and they are overlapped due to the influence of coordination number. Therefore, it is evident that the application of (^{119}Sn) values for the prediction of the coordination mode around the tin atom in mono-n-butyltin compounds (and probably in other monoorganotin compounds as well)

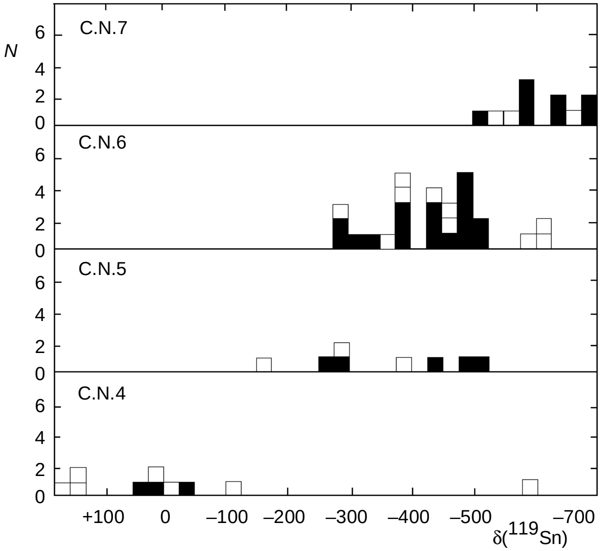

FIG. 1

The δ(^{119}Sn) values of mono-n-butyltin(IV) compounds (intervals of 25 ppm) corresponding to coordination numbers of tin atom of 4–7. *N* number of (^{119}Sn) values; ❐ data from this paper, ■ literature data must be done carefully, especially within the ranges where the values of $\delta(^{119}Sn)$ for different coordination numbers mutually overlap. Nevertheless, in a first approximation we can consider the $\delta(^{119}Sn)$ values ranging from +160 to –120 ppm (except for *III*, vide infra) as characteristic of the four-coordinated (pseudotetrahedral) mono-n-butyltin compounds. The mono-n-butyltin complexes with five-coordinated tin atom possess the values of $\delta(^{119}Sn)$ within a relatively broad range from –150 to –530 ppm, those with six-coordinated tim atom from –270 to –620 ppm and those with the coordination number seven from about –500 to –700 ppm.

The uncertainities in the evaluation of the coordination number of central atom in mono-n-butyltin compounds, using the parameter of $\delta(^{119}Sn)$, can be reduced by an additional application of some parameters of ^{13}C NMR spectra.

In the series of the compounds *I*, *II* and *III*, the $\delta(^{119}Sn)$ values are shifted substantially upfield on going from *I* to *III*; the compound *III* reveals an extraordinary $\delta(^{119}Sn)$ value (–577.8). Simultaneously the values of $^1J(^{119}Sn,^{13}C)$ are remarkably decreasing in this series. Therefore, it is evident that the upfield shift of $\delta(^{119}Sn)$ value in *III* (and to a some extent in *II*) is not associated with an increasing coordination of the tin atom because in this case both parameters should increase. We assume that the observed increase in $\delta(^{119}Sn)$ parameter is due to so called "heavy atom effect", which overlaps all other effects (unlike at the compounds as Bu_3SnI or Bu_2SnI_2) (refs[2,3]) including the effect of coordination. As both parameters $\delta(^{119}Sn)$ and $^1J(^{119}Sn,^{13}C)$ change only slightly with temperature and concentration of deuteriochloroform solutions, we cannot expect a significant self-association of *I–III*. Thus, these compounds can be considered to be tetracoordinated. Therefore, in solutions of nonpolar (non-coordinating) solvents these compounds exist in the form of simple tetrahedral molecules.

This conclusion is also supported by the $\delta(^{119}Sn)$ values obtained by measuring solutions of *I* and *II* in a coordinating solvent. A significant upfield shift caused by a coordination with hexadeuteriodimethyl sulfoxide, being 463.0 and 489.3 ppm, respectively, into the region of $\delta(^{119}Sn)$ values characteristic of six-coordinated mono-n-butyltin compounds gives the evidence for the formation of complexes of the type $BuSnX_3 . 2\ S$ (X = Cl or Br, S is a solvent molecule) as a result of the equilibrium (*1*)

$$BuSnX_3 + 2\ S \rightleftarrows BuSnX_3 . 2\ S \qquad (1)$$

which is shifted significantly in favour of the reaction products. The formation of compounds $BuSnX_3 . 2\ S$ has been confirmed several times in literature (e.g. refs[31–34]). The presence of the six-coordinated complex $BuSnCl_3 . 2\ (CD_3)_2SO$ in a surplus of the solvent manifests also the similarity of its $\delta(^{119}Sn)$ value with that for the complex anion of $[BuSnCl_5]^-$ in *IV*.

Tetrahedral coordination of the tin atom can be assigned also to the simple molecules of *V*–*VII*. Their δ(^{119}Sn) values in deuteriochloroform solutions (from +158.9 to –12.3 ppm) belong indicatively into the region of δ(^{119}Sn) values characteristic of mono-n-butyltin compounds with the four-coordinated tin atom. Similarly, small changes in the value of δ(^{119}Sn) of the *V* when replacing non-coordinating solvent (deuteriochloroform) by coordinating one (pentadeuteriopyridine was used because *V* is practically insoluble in hexadeuteriodimethyl sulfoxide), is evidently due to the existence of the equilibrium of the type (*1*) which is significantly shifted to the substrate side in this case. This observation is in agreement with the well-known inability of the organotin(IV) compounds containing only –SR arrangement as polar groups towards the formation of donor–acceptor complexes[35]. The presence of chlorine atom in *VI* besides of SCH_2CH_2S has changed acceptor properties of the central tin atom, compared with that in *V*, in such an extent that also this compound forms pseudooctahedral complex with two molecules of hexadeuteriodimethyl sulfoxide, according to the Eq. (*1*).

The values of δ(^{13}C) of carbon atoms in the carboxyl group in *VIII*–*X* are practically identical with that for acetate group (181.7 ppm) (ref.[36]), but very different from that of the organic esters of acetic acid (about 170 ppm) (ref.[37]). This fact gives the evidence for the bidentate binding of the carboxylic group in the coordination polyhedra of these compounds. Moreover, in ^{13}C NMR spectra of *VIII*–*X* only one series of the NMR signals has been observed, which gives evidence for the equivalence of acetate groups in these compounds, at least on the NMR time scale. When the chlorine atoms are replaced step-by-step by acetate groups in the above-mentioned compounds the coordination number of the central atom increases, which is in agreement with an upfieled shift of δ(^{119}Sn) values and an increase in the $^1J(^{119}\mathrm{Sn},^{13}\mathrm{C})$ values. Chemical shifts δ(^{119}Sn) and $^1J(^{119}\mathrm{Sn},^{13}\mathrm{C})$ values increase nearly linearly, within the experimental error, with increasing coordination number in the series of $BuSnCl_{3-n}(CH_3COO)_n$ compounds for n = 0–3. If these compounds exist in deuteriochloroform as monomers (what we can assume from the fact that their ^{13}C and ^{119}Sn NMR parameters do not change neither with the temperature nor with the concentration significantly, and also from the analogy of these compounds with tri- and di-n-butyltin compounds[2,3]), then the molecule of *VIII* assumes a distorted trigonal bipyramide geometry, *IX* is pseudooctahedral and *X* has a geometry of a distorted pentagonal bipyramide.

Practically the same conclusion can be deduced from the analysis of the NMR data in the two other series of $BuSnCl_{3-n}(edtc)_n$ (n = 0–3) (*I*, *XI*, *XII* and *XV*) and $BuSnBr_{3-n}(edtc)_n$ (n = 0–3) (compounds *II* and *XIII*–*XV*). Also in these cases both the values of δ(^{119}Sn) and $^1J(^{119}\mathrm{Sn},^{13}\mathrm{C})$ increase with an increasing parameter n (i.e., increasing number of edtc groups), giving thus the evidence for an increasing coordination number but the linearity of the observed changes is not so pronounced. The reason for it could be caused by the fact that the values of δ(^{13}C) in the CS_2 group of the edtc ligands increase with increasing number *n*, as a result of an increasing bonding strength of the CS_2 group due to the formation of chelate ring with the tin atom[35].

The series of compounds $BuSnCl_{3-n}(oxin)_n$ (*I*, *XVI* and *XVIII*) and $BuSnBr_{3-n}$ $(oxin)_n$ (*II*, *XVII* and *XVIII*) are not complete; we were not successful in the preparation of pure compounds $BuSnCl_2(oxin)$ and $BuSnBr_2(oxin)$. Nevertheless, it is evident in this case from the $\delta(^{119}Sn)$ and $^1J(^{119}Sn,^{13}C)$ parameters, that *XVI* and *XVII* are hexa-coordinated pseudooctahedral molecular complexes. The compound *XVIII* can be evidently considered as a complex with the coordination number of tin atom equal to seven; its shape corresponds to a distorted pentagonal bipyramid. The complex *XIX* seems to be also pseudooctahedral, regardless of relatively low values of the corresponding ^{119}Sn and ^{13}C NMR spectral parameters, which are, after all, typical for complexes with thiooxinate ligands[35,38–40].

The ^{13}C and ^{119}Sn NMR spectral data of a heteroleptic $[BuSnX(chel)_2]$ (where X = Cl or Br, chel = AcO, edtc, oxin or txin) octahedral complexes and pentagonal-bipyramid homoleptic $[BuSn(chel)_3]$ complexes are in some cases in contradiction with results obtained from their X-ray diffraction analysis. For example, in the crystal structure of *XV* (ref.[41]) and of further compounds $RSn(edtc)_3$ (ref.[12]) at least two types of chelated edtc ligands are presented, whereas their ^{13}C NMR spectra show only one set of NMR signals for all carbon atoms. From this it follows that the compounds of the type $RSnCl(chel)_2$ (ref.[41]) do not contain equivalent chelate groups in the solid state. The conforming results were obtained for complexes of *XVI* and *XVII*, where two sets of signals were observed in their ^{13}C NMR spectra giving the evidence for the presence of two different chelate groups. The complexes with the chelated groups of AcO, txin and edtc usually manifest equivalency of ligands in their ^{13}C NMR spectra. Several influences can cause the observed behaviour, the differentiation among them is the subject of our present studies.

CONCLUSION

From the results discussed above it can be concluded that the knowledge of the parameters of ^{13}C and ^{119}Sn NMR spectra in mono-n-butyltin compounds, especially of $\delta(^{119}Sn)$ chemical shifts and $^1J(^{119}Sn,^{13}C)$ coupling constants and their combination is very important for the prediction of the geometry of Sn(IV) coordination polyhedra of these compounds in the solutions of various solvents. Contrary to tri- and di-n-butyl compounds (and, in fact, to generally all tri- and diorganotin(IV) compounds) the coordination numbers in mono-n-butyltin compounds cannot be estimated only from the $\delta(^{119}Sn)$ values without the knowledge of $^1J(^{119}Sn,^{13}C)$ coupling constants and further ^{13}C NMR spectral parameters.

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