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**^{119}Sn , ^{15}N , ^{13}C AND ^1H NMR STUDY OF THE
INTRAMOLECULAR Sn-N DONOR-ACCEPTOR
INTERACTION IN SOME [8-(DIMETHYL-
AMINO)-1-NAPHTHYL]STANNANES**

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A set of six [8-(dimethylamino)-1-naphthyl]stannanes $R^1R^2R^3\text{Sn}[8-(\text{CH}_3)_2\text{N}-1-\text{C}_{10}\text{H}_8]$, where R^1 , R^2 and R^3 are $1-\text{C}_4\text{H}_9$, Cl, Br or $[8-(\text{CH}_3)_2\text{N}-1-\text{C}_{10}\text{H}_8]$, the same or different, was prepared and their ^{119}Sn , ^{15}N , ^{13}C and ^1H NMR spectra were studied. The existence of intramolecular donor-acceptor interaction between the nitrogen and tin atoms was demonstrated as a consequence of characteristic

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Introduction

The existence of the Sn-N intramolecular interaction (and also its measure and stability) in various [8-(dimethylamino)-1-naphthyl]stannanes $R^1R^2R^3SnL$, where $L = [8-(CH_3)_2N-1-C_{10}H_6]$, R^1 , R^2 and R^3 are the same or different organic substituents (1-C₄H₉ (Bu) and L) or halogene atoms, and, hence, formation of corresponding five-membered azastannacycles (C,N-chelates), has been proved many times in the solid state by using X-ray single crystal diffraction techniques [1–5]. The systematic and very extensive research by van Koten's working group based on the synthesis of numerous well-selected compounds and their detailed examination using ¹H, ¹³C and ¹¹⁹Sn NMR spectroscopy suggests that this interaction remains preserved in many cases also in isolated particles of these compounds in solutions.

It is evident that the formation of this intramolecular interaction induces deep changes in NMR spectra parameters of all the nuclei as a consequence of changes in bonding and geometrical situation in molecules of compounds. It is possible to expect that the most important changes will be observed in NMR parameters of nuclei directly involved in the Sn-N interaction and nuclei in their nearest neighbourhood. However, as far as we know, there exists an only limited pieces of evidence of such interaction based on the tin and nitrogen NMR parameters [6–13].

In the present paper it will be reported on the existence, its measure and structural consequences of the Sn-N interaction in the set of six [8-(dimethylamino)-1-naphthyl]stannanes $R^1R^2R^3SnL$, where R^1 , R^2 and R^3 are organic substituents Bu or L and/or halogen atoms Cl, Br, I, the same or different, (Table I), in the solution of non-polar solvent (deuteriochloroform) on the basis of ¹¹⁹Sn, ¹⁵N, ¹³C and ¹H NMR spectra. As the natural abundance of ¹⁵N nucleus is very low (0.37%), more soluble [8-(dimethylamino)-1-naphthyl](1-butyl)(halogeno)stannanes were studied while van Koten's school described methyl and phenyl analogues.

Experimental

General

All the reactions mentioned were carried out in dry argon atmosphere using standard Schlenck techniques. Diethylether, benzene, toluene and *n*-hexane were dried over sodium and distilled, chloroform was dried over P₂O₅ and distilled from LiAlH₄. All other reagents (1-aminonaphthalene, *n*-butyllithium and dimethyl sulphate) were obtained from commercial sources (Merck) and were used without further

purification. 1-Dimethylamino-naphthalene (LH) was prepared from 1-amino-naphthalene, dimethyl sulphate and sodium hydrogencarbonate in water solution [14]. The (8-dimethylamino)-1-naphthyllithium etherate was prepared from 1-dimethylamino-naphthalene (LH) and *n*-butyllithium in the diethylether / *n*-hexane (1:1) mixture [15] ($\delta(^7\text{Li}) = 4.5$ ppm, $\delta(^{15}\text{N}) = -369.0$ ppm). The compounds prepared are unstable (in light and air) and decompose to dark green or deep blue oils.

Preparation of Compounds Studied

Bu₃SnL (1). (8-Dimethylamino)-1-naphthyllithium etherate (7.2 g, 0.028 mol) in 25ml of benzene was slowly (30 min.) added to a solution of *Bu₃SnCl* (9 g, 0.027 mol) in 25 ml of benzene and then stirred. After 30 min of stirring the pale brown precipitate was filtered off, washed twice with 20 ml chloroform, and the combined filtrates were evaporated *in vacuo*. The dark yellow residue was the distilled under reduced pressure (155-160 °C/ 50 Pa) to give (1) as a yellowish oil (7.05 g, 70%).

Bu₂SnL₂ (2). (8-Dimethylamino)-1-naphthyllithium etherate 7.2 g (0.028mol) in 25ml of benzene was slowly (30 min) added to a solution of *Bu₂SnCl₂* (4 g, 0,013 mol) in 25 ml benzene. After 30 min stirring the brown precipitate was filtered off, washed twice with 20 ml chloroform, and the combined filtrates were evaporated *in vacuo*. The dark yellow residue was then distilled under reduced pressure (192-195 °C/ 50 Pa) to give (2) as a yellow oil (4.35 g, 57%).

Bu₂SnClL (3), *Bu₂SnBrL* (4) and *Bu₂SnIL* (5). The compounds were prepared as was compound (1) from (8-dimethylamino)-1-naphthyllithium etherate and the corresponding dibutyltin(IV)dihalides (yields 65 - 70%).

SnCl₂L₂ (6). (8-Dimethylamino)-1-naphthyllithium etherate (7.2g, 0.028 mol) in 25 ml benzene was slowly (30 min) added to a solution of *SnCl₄* (3.4 g, 0.013 mol) in 25 ml benzene and then stirred. After 30 min stirring, the brown precipitate was filtered off, washed twice with 20 ml chloroform, and the combined filtrates were evaporated *in vacuo*. Resulting brown solid was recrystallized from boiling toluene to give white crystals (4.7 g, 70%).

Identification of Compounds

The identity of the compounds prepared was confirmed by elemental analysis (Table I) and by analysis of their ¹¹⁹Sn, ¹⁵N, ¹³C and ¹H NMR spectra (Tables II – IV). The compounds 1 – 6 were characterised by basic physical constants (Table I).

Table I Analytical and physical data of compounds 1 – 6

Compound	R ¹	R ²	R ³	Formula/M.w., g mol ⁻¹
1 Bu ₃ SnL ^(a)	Bu	Bu	Bu	C ₂₄ H ₃₉ NSn 461.28
2 Bu ₂ SnL ₂	Bu	Bu	L	C ₄₀ H ₆₀ N ₂ Sn 561.40
3 Bu ₂ SnLCl	Bu	Bu	Cl	C ₂₀ H ₃₀ NSnCl 439.62
4 Bu ₂ SnLBr	Bu	Bu	Br	C ₂₀ H ₃₀ NSnBr 484.07
5 Bu ₂ SnLI	Bu	Bu	I	C ₂₀ H ₃₀ NSnI 531.07
6 SnL ₂ Cl ₂	Cl	Cl	L	C ₂₄ H ₂₄ N ₂ SnCl ₂ 518.07

Compound	Found / (Calculated), w.%					m.p./ (b.p.) °C / Pa
	H	C	N	Sn	Other	
1 Bu ₃ SnL ^(a)	62.41 (62.49)	8.65 (8.74)	2.91 (3.04)	25.67 (25.73)	-	155 - 160 / (50)
2 Bu ₂ SnL ₂	68.39 (68.46)	7.85 (7.90)	2.31 (2.49)	20.94 (21.14)	-	190 - 195 / (50)
3 Bu ₂ SnLCl	54.55 (54.64)	7.02 (7.11)	3.05 (3.19)	26.91 (27.00)	Cl: 7.98 (8.06)	181 - 183 / (50)
4 Bu ₂ SnLBr	49.57 (49.63)	6.38 (6.45)	2.74 (2.89)	24.47 (24.52)	Br: 16.43 (16.51)	175 - 180 / (50)
5 Bu ₂ SnLI	45.12 (45.23)	5.78 (5.88)	2.51 (2.64)	22.24 (22.35)	I: 23.82 (23.90)	200 - 205 / (50)
6 SnL ₂ Cl ₂	55.59 (55.64)	5.00 (5.06)	2.61 (2.70)	22.80 (22.91)	Cl: 13.81 (13.92)	323 - 326

a) For structure of compounds 1 – 6 see Table II

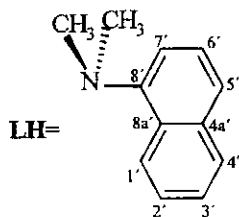
NMR Spectra

The ¹¹⁹Sn, ¹⁵N, ¹³C and ¹H NMR spectra were measured on a Bruker AMX 360 instrument at 134.28, 36.56, 90.56 and 360.13 MHz, respectively, using a 5 mm

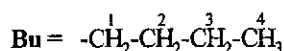
Table II ^1H NMR data of compounds LH and 1 – 6

$\delta(^1\text{H})$, ppm	Compound ^(a,b)						
	LH	1	2	3	4	5	6
H(1')	7.26	-	-	-	-	-	-
H(2') ^(c)	8.16	7.71 (61.4)	7.81 (71.0)	8.55 (75.9)	8.58 (77.3)	8.62 (78.5)	8.67 (127.0)
H(3')	7.20	7.26	7.35	7.50	7.53	7.50	7.77
H(4')	7.56	7.55	7.65	7.78	7.80	7.78	7.99
H(5')	7.29	7.39	7.46	7.64	7.68	7.68	7.75
H(6')	7.12	7.18	7.17	7.38	7.42	7.42	7.47
H(7')	6.73	7.02	6.99	7.35	7.39	7.37	7.37
N(CH ₃) ₂ ^(d)	2.53 (133.1)	2.48 (136.1)	2.73 (136.2)	2.70 (138.8)	2.75 (137.7)	2.75 (136.9)	2.73 ^(e) 1.80 ^(e) (136.9)
H(1)	-	1.02	1.48	1.42	1.46	1.45	-
H(2)	-	1.48	2.01	1.61	1.60	1.60	-
H(3)	-	1.28	1.24	1.25	1.25	1.24	-
H(4)	-	0.82	0.72	0.75	0.76	0.75	-

a)



b)

c) $^3\text{J}(^{119}\text{Sn}, ^1\text{H})$ in parentheses d) $^1\text{J}(^{13}\text{C}, ^1\text{H})$ in parentheses e) anisochronous protons

tunable broad-band probe at 300 K. The solutions were obtained by dissolving approximately 200 mg the substance in 0.5 ml deuteriochloroform.

The reported ^1H and ^{13}C chemical shifts are related to internal tetramethylsilane ($\delta = 0.00$ ppm), the ^{15}N and ^{119}Sn chemical shifts are relative to external nitromethane and tetramethylstannane, respectively, placed in coaxial capillaries ($\delta = 0.00$ ppm). Positive values denote downfield shifts. The $\text{J}(^{119}\text{Sn}, ^{15}\text{N})$ coupling

constants were read from the ^{15}N NMR spectra measured at a digital resolution better than 0.5 Hz/point. The 2D NMR spectra were measured conventionally using pulse sequence microprograms supplied by the manufacturer of the spectrometer.

Results and Discussion

^{119}Sn and ^{13}C NMR spectra

X-ray single crystal analysis of the phenyl analogue of compound *1* showed a very distorted trigonal bipyramidally coordinated central tin atom with the coordinating nitrogen atom and one of the phenyl groups in axial positions [16]. However, the Sn-N bond distance of 2.841 Å seems to be rather long, so that donor-acceptor interaction must be very weak. Moreover, the solid-state structure is not necessarily representative of the structure in the solution. The ^{119}Sn chemical shift value of compound *1* in the solution of non-polar solvent (deuteriochloroform) is very close to that of its methyl analogue (-50.0 ppm) [17] and both values are only slightly shifted upfield (less than 20 ppm) compared with that of unsubstituted trimethyl-1-naphthyl-stannane [18]. This fact agrees with the only very weak (if any) Sn-N donor-acceptor interaction in compound *1* in the deuteriochloroform solution.

The very similar weak Sn-N bonding interaction is assumed also in compound *2* on the basis of the slight upfield shift (ca. 23 ppm) of its $\delta(^{119}\text{Sn})$ value compared with that of dimethyldi(1-naphthyl)stannane (-65.0 ppm [17]).

Coupling constants $^1\text{J}(^{119}\text{Sn},^{13}\text{C}(1))$ and $^1\text{J}(^{119}\text{Sn},^{13}\text{C}(1'))$ of compounds *1* and *2* (Tables III and IV) are more typical of tetrahedral arrangement in the bonding environment of the central tin atoms than that of trigonal bipyramidal [19,20]. This is confirmed by average C(1)-Sn-C(1) and C(1')-Sn-C(1') angle values (ca. 110° (compound *1*), ca. 111° and 109°, respectively (compound *2*), their calculation being based on $^1\text{J}(^{119}\text{Sn},^{13}\text{C}(1))$ and $^1\text{J}(^{119}\text{Sn},^{13}\text{C}(1'))$ data [21,22] (114.4, 101.1 and 103.4°, average 106.3°, in the phenyl analogue of the compound in solid state [2]). Considering a weak Sn-N interaction, the shapes of the coordination polyhedra around the central tin atom in compounds *1* and *2* in solution of the non-polar solvent can better be formulated as slightly distorted (capped and bicapped, respectively) tetrahedra as trigonal bipyramides.

The direct comparison of $\delta(^{119}\text{Sn})$ chemical shift values of compounds *3*, *4*, *5* and *6* with those of unsubstituted analogues could not be made because $\delta(^{119}\text{Sn})$ values of butyl(halogeno)naphthylstannanes are not available. Nevertheless, the $\delta(^{119}\text{Sn})$ values of triorganotin(IV) compounds *3*, *4*, *5* all display marked (150 - 200 ppm) upfield shifts in comparison with the corresponding tributyl(halogeno)-stannanes [20], the $\delta(^{119}\text{Sn})$ value of compound *6* being upfield shifted as compared with diphenyldichlorostannane (representative of diaryldichlorostannanes) by more than 260 ppm [23]. All these changes reflect the general tendency of tri- and di-

Table III NMR spectral data of compounds LH and 1 – 6

Compound	$\delta(^{15}\text{N})$ ppm	$\delta(^{119}\text{Sn})$ ppm	$J(^{119}\text{Sn}, ^{15}\text{N})$	$\delta(^{13}\text{C}), \text{ppm} / ^1J(^{119}\text{Sn}, ^{13}\text{C}), \text{Hz}$				
				C(1)	C(2)	C(3)	C(4)	C(NCH ₃)
LH	-345.85	-	-	-	-	-	-	44.54
1	-344.48	-50.4	21.7	11.71 (358.7)	29.34 (18.1)	27.64 (62.1)	13.72 (b)	47.18 (b)
2	-348.09	-87.8	23.6	14.21 (365.6)	29.10 (11.9)	27.68 (68.5)	11.70 (b)	47.70 (b)
3	-345.61	-37.8	66.1	19.10 (489.9)	27.49 (30.7)	25.96 (84.6)	12.91 (b)	47.82 (b)
4	-344.68	-28.1	63.5	20.37 (478.0)	27.96 (30.7)	26.15 (86.7)	13.12 (b)	48.19 (b)
5	-343.49	-20.5	61.9	21.54 (463.4)	28.10 (31.0)	25.77 (86.9)	12.96 (b)	48.12 (b)
6	-348.77	-236.26	(b)	-	-	-	-	52.10 ^(c) (b) 47.56 ^(c) (b)

- a) For structure and numbering of compounds 1 – 6 see Table II
 b) Not found
 c) anisochronous carbons

diorgano(halogeno)-stannanes to be five- and six-coordinate, respectively, in the presence of donor atoms.

The C(1)–Sn–C(1) angles in compounds 3, 4, and 5 (121 – 123°) calculated [21] on the basis of $^1J(^{119}\text{Sn}, ^{13}\text{C})$ coupling constant values correspond to the trigonal bipyramidal vicinity of central five-coordinated tin atom with both C(1) and C(1') carbon atoms in equatorial plane and chlorine and nitrogen atoms in axial positions. It should be noted here that distorted trigonal bipyramidal coordination of tin atoms was found in crystals of some analogues of compounds 3 – 5 [5] by means of X-ray diffraction.

Based on $\delta(^{119}\text{Sn})$ and $^1J(^{119}\text{Sn}, ^{13}\text{C}(1'))$ values, the central tin atom in compound 6 is six-coordinated with both carbon atoms C(1') in *trans* position of distorted octahedron, the calculated [22] C(1')–Sn–C(1') angle being approximately 152° (153.9° in the crystals of the bromine analogue [4]).

¹⁵N NMR Spectra

As a result of the increasing strength of Sn-N interaction and of changes in geometry of co-ordination polyhedra in set of four- < five- < six- coordinated compounds (e.g.

Table IV ^{13}C NMR data of compounds LH and *l* – *6*

$\delta(^{13}\text{C}), \text{ppm} /$ $(^a\text{J}(^{119}\text{Sn}, ^{13}\text{C}), \text{Hz})$	Compound ^(b)						
	LH	<i>l</i>	<i>2</i>	<i>3</i>	<i>4</i>	<i>5</i>	<i>6</i>
C(1')	124.71	137.93 (486.4)	140.36 (583.3)	134.72 (640.78)	134.15 (622.4)	132.75 (596.3)	134.80 (1261.5)
C(2')	123.94	136.11 (31.6)	135.25 (35.1)	136.43 (33.8)	137.67 (35.8)	139.01 (36.3)	133.89 (52.3)
C(3')	125.32	125.43 (54.34)	125.73 (44.9)	126.28 (67.7)	126.59 (67.5)	126.31 (67.6)	127.14 (122.5)
C(4')	128.04	128.46 (11.8)	128.16 (12.7)	128.37 (14.2)	128.67 (14.6)	128.63 (15.1)	130.28 (22.2)
C(4a')	128.63	135.02 (32.6)	134.99 (19.4)	133.44 (45.6)	133.67 (45.1)	133.55 (43.6)	134.32 (b)
C(5')	122.53	125.39 (b)	125.35 (b)	126.34 (b)	126.65 (b)	126.35 (b)	127.23 (b)
C(6')	125.48	125.14 (b)	125.09 (b)	124.99 (b)	125.23 (b)	125.17 (b)	125.97 (b)
C(7')	113.58	114.93 (5.6)	116.44 (b)	115.81 (8.7)	115.88 (8.5)	115.61 (8.3)	118.43 (9.7)
C(8')	150.53	152.61 (13.70)	153.09 (15.8)	148.94 (22.0)	148.97 (21.4)	148.45 (20.6)	148.78 (23.3)
C(8a')	134.63	134.71 (21.9)	134.87 (11.8)	133.18 (40.4)	133.28 (38.8)	132.61 (36.2)	131.89 (76.4)

a) For structure and numbering of compounds *l* – *6* see Table II

b) Not found

l < *3* < *6*), significant changes in ^{15}N NMR parameters would be expected also. The magnitude and sign of the $\Delta\delta(^{15}\text{N})$ coordination shifts (the difference between the chemical shift $\delta(^{15}\text{N})$ value of coordinated ligand and that of free one) similarly as a $\Delta\delta(^1\text{H})$ protonation shifts (difference between the protonated and unprotonated compound with a lone electron pair) depend among others on the type of the hybridization of the nitrogen atoms [24,25]. While protonation of aliphatic amines and also their participation in coordination sphere of the main group metals induce downfield shifts, sp^2 and sp hybridized nitrogen atoms are in general opposite in direction to the former. Both types of such changes were observed in our previous papers. While the formation of chelate organotin(IV) 8-quinolinolato- and 8-quinolinethiolato complexes are characterized by upfield coordination shift of $\delta(^{15}\text{N})$ [8–13], downfield coordination shift was observed as a result of increasing strength of Sn-N interaction in C,N chelate [2-(dimethylaminomethyl)phenyl]butyltin(IV)

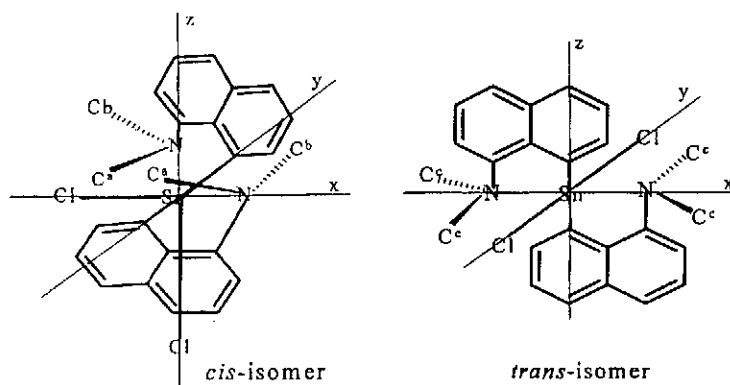


Fig.1 Two possible geometrical isomers of compound 6

complexes [7].

The $\Delta\delta(^{15}\text{N})$ coordination shift values of the compounds studied are practically useless for an assessment of existence and measure of Sn-N interaction because of their both positive and negative signs and also of very narrow interval of their occurrence (from +2.36 to -2.92 ppm). It seems to, that this ambiguous behaviour of the nitrogen atom of the dimethylamino group bonded directly to the naphthalene ring is a consequence of two opposite influences, that of sp^3 hybridization of nitrogen atom on one hand and the delocalisation of its lone pair into an aromatic system [26] on the other hand.

In contrast to parameter $\Delta\delta(^{15}\text{N})$, coupling constant $J(^{119}\text{Sn}, ^{15}\text{N})$ values play much more important role for estimation of the Sn-N interaction in the compounds studied. These values increase from ca 20 Hz to more than 60 Hz on going from tetraorganotin(IV) compounds (1 and 2) with very weak interaction to five-coordinated complexes (3 - 5) with distinctly bonding donor-acceptor interaction forming slightly distorted trigonal bipyramide coordination vicinity of the central tin atom with three carbon atoms in equatorial plane and halogen and nitrogen atoms in axial positions. Unfortunately, the expected further increase in $J(^{119}\text{Sn}, ^{15}\text{N})$ with increasing coordination number of central tin atom (e.g. comparison of $J(^{119}\text{Sn}, ^{15}\text{N})$ values of compounds 2 and 6) is impossible – the $J(^{119}\text{Sn}, ^{15}\text{N})$ value of compound 6 was not measured because of its low solubility in deuterio-chloroform. However, *cis*-positions of nitrogen and chlorine atoms in octahedral shape of coordination polyhedron of this compound is very well documented by the presence of two signals for CH_3 groups of dimethylamino substituent in ^{13}C and ^1H NMR spectra (see Tables II and III, Fig.1).

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