Journal of Organometallic Chemistry, 339 (1988) 61–68
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

^{13}C and ^{119}Sn NMR spectra of divinyltin(IV) compounds

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(Received July 14th, 1987)

Abstract

The ^{13}C and ^{119}Sn NMR spectra of a set of divinyltin(IV) compounds in coordinating and non-coordinating solvents have been studied. The obtained results are discussed in terms of relations between the values of NMR spectral parameters and the geometries of coordination polyhedra of molecules of the divinyltin(IV) compounds and their complexes.

Introduction

In previous papers [1–3] we applied ^{13}C and ^{119}Sn NMR spectral parameters to the description and differentiation of the three basic structural types of the coordination polyhedra of central tin atom in triorganotin(IV) compounds and their complexes in various solvents. There are good linear correlations between the parameters which reflect the structure of triorganotin(IV) compounds ($\delta(^{119}Sn)$ and $^1J(^{119}Sn, ^{13}C)$) for simple R_3SnX compounds and $R_3SnX \cdot L$ complexes [2–4] (R = organic substituent, X = polar group, L = Lewis base, e.g. the molecule of a solvent) having the same structure but only different R substituents. These correlations are due to a mutual similarity of coordination polyhedra of triorganotin(IV) compounds and also because of a slight deviation from ideal shape (tetrahedral or trigonal-bipyramidal). In spite of the larger variability of structural types of diorganotin(IV) compounds and their complexes, we found [5] relatively sharply defined ranges of $\delta(^{119}Sn)$ values which are characteristic of tetra-, penta- or hexa-coordinated central tin atom in di-n-butyltin(IV) compounds. $^1J(^{119}Sn, ^{13}C)$ values permitted us to elucidate the C–Sn–C angles in the n-Bu_2Sn group and thus

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0022-328X/88/$03.50

the shapes of their coordination polyhedra and the degree to which the polyhedra were distorted.

Our continuing interest in the relations between the structure of organotin(IV) compounds and their complexes in solutions of various solvents and the parameters of their NMR spectra raised the question of whether the relations observed in di-n-butyltin(IV) compounds have a more general validity, and whether there are similar mutual relations between NMR spectral parameters of diorganotin(IV) compounds with various substituents like those in triorganotin(IV) compounds.

We have studied ^{13}C and ^{119}Sn NMR spectra of a series of divinyltin(IV) compounds and their complexes which contains all of the known geometries that the coordination polyhedra of diorganotin(IV) compounds can take and have compared them with the ^{13}C and ^{119}Sn NMR spectra of di-n-butyltin(IV) compounds. The previous interest in the ^{13}C and ^{119}Sn NMR spectra of the compounds containing $R^1R^2C{=}C(R^3)Sn$ group, either with a simple or substituted vinyl group (R^1, R^2, R^3 are organic substituents or hydrogen atoms), was limited only to tetraorganotin(IV) compounds. Compounds which have a polar substituent, in addition to a vinyl group, bonded to the tin atom have been studied only sporadically without specific purposes [6,7]. The complexes of vinyltin(IV) compounds have to our knowledge not yet been studied.

Results and discussion

The ^{13}C and ^{119}Sn parameters of the NMR spectra of the studied compounds are listed in Table 1.

Chemical shifts $\delta(^{119}Sn)$

The values of the chemical shift $\delta(^{119}Sn)$ of the studied compounds lie in the relatively broad range from +16.3 for $Vi_2Sn(SBu\text{-}n)_2$ to −519.0 for the complex $Vi_2Sn(OCOPh)_2 \cdot 2dmso\text{-}d_6$ (vide infra). These values depend on the coordination of the central tin atom in common with those for all organotin(IV) compounds. This is evident from Fig. 1, where the mutual relations between the $\delta(^{119}Sn)$ of divinyltin(IV) compounds and $\delta(^{119}Sn)$ of di-n-butyltin(IV) compounds studied previously [5] is plotted. This relation is linear, which is shown by the linear regression analysis obtained by a method of least-squares:

$$\delta(^{119}Sn)_{Vi_2Sn} = (1.03 \pm 0.04)\delta(^{119}Sn)_{n\text{-}Bu_2Sn} - (155 \pm 8.77) \quad (1)$$
$$N = 14,\ r = 0.991$$

(The values of $\delta(^{119}Sn)$ of the di-n-butyltin(IV) compounds, and their complexes, used for the correlation (1) were taken from ref. 5 combined with our unpublished results.)

The relationships between the chemical shift $\delta(^{119}Sn)$ and the coordination of central tin atom in di-n-butyltin(IV) compounds was used together with the linear correlation (1) of $\delta(^{119}Sn)$ for both types of compounds we estimated rough limits for the $\delta(^{119}Sn)$ values of divinyltin(IV) compounds, and their complexes, for various coordination numbers of the central tin atom. From the ranges of $\delta(^{119}Sn)$ values of di-n-butyltin(IV) compounds with four-coordinated (+200 to −60 ppm), five-coordinated (−90 to −190 ppm) and six-coordinated (−210 to −400 ppm) tin atoms, we obtained (for divinyltin(IV) compounds) the $\delta(^{119}Sn)$ values +20 to

Table 1

^{13}C and ^{119}Sn NMR spectral data of divinyltin(IV) compounds

Compound [a]	Solvent	$\delta(^{119}Sn)$ (ppm)	$\delta(^{13}C)$ (ppm)		$^1J(^{119}Sn, ^{13}C)$ (Hz)
			C(1)	C(2)	
Vi_2SnCl_2 (**1**)	$CDCl_3$	−30.2	134.12	139.53	750.7
	dmso-d_6	−394.1	152.52	129.23	1469.7
Vi_2SnBr_2 (**2**)	$CDCl_3$	−76.4	134.12	139.19	688.7
	dmso-d_6	−407.9	152.67	129.56	1445.3
Vi_2SnI_2 (**3**)	$CDCl_3$	−254.7	133.82	138.65	604.2
	dmso-d_6	−447.7	148.81	132.44	1453.9
$Vi_2Sn(SBu\text{-}n)_2$ (**4**)	$CDCl_3$ [b]	16.3	134.90	137.29	571.3
	dmso-d_6 [c]	−24.2	138.34	136.05	636.0
$Vi_2Sn(OCOMe)_2$ (**5**)	$CDCl_3$ [d]	−309.5	135.68	138.11	982.7
	dmso-d_6 [e]	−510.2	147.70	130.10	1463.6
$Vi_2Sn(OCOPh)_2$ (**6**)	$CDCl_3$ [f]	−317.7	136.46	138.07	1010.7
	dmso-d_6 [g]	−519.0	147.84	130.39	1492.9
$Vi_2Sn(NCS)_2$ (**7**)	dmso-d_6 [h]	−531.9	147.24	131.45	1581.1
$Et_4N^+ Vi_2SnCl_3^-$ (**8**)	CD_3NO_2 [i]	−253.6	148.53	135.76	1041.3
$Vi_2Sn(oxin)_2$ (**9**)	$CDCl_3$	−394.1	147.08	132.26	897.5
$Vi_2Sn(edtc)_2$ (**10**)	$CDCl_3$ [j]	−471.7	145.45	130.65	956.9

[a] Abbreviations: Vi = $\overset{2}{C}H_2{=}\overset{1}{C}H-$, dmso-$d_6$-hexadeuteriodimethyl sulphoxide, oxin = 8-hydroxyquinolinate, edtc = *N,N*-diethyldithiocarbamate. [b] $\delta(^{13}C)$: 36.21 ($J(^{119}Sn, ^{13}C)$ 17.9 Hz), 26.90 ($J(^{119}Sn, ^{13}C)$ 15.8 Hz), 21.99, 13.70. [c] $\delta(^{13}C)$: 36.09, 26.59, 21.28, 13.52. [d] $\delta(^{13}C)$: 181.49(COO), 20.32(CH_2). [e] $\delta(^{13}C)$: 176.90(COO), 21.23(CH_3). [f] $\delta(^{13}C)$: 173.59(COO), 129.29(*i*), 130.27(*o*), 128.37(*m*), 133.68(*p*). [g] $\delta(^{13}C)$: 169.24(COO), 131.13(*i*), 129.57(*o*), 128.61(*m*), 132.98(*p*). [h] $\delta(^{13}C)$: 137.88(NCS), $\delta(^{14}N)$: −212 ($w_{1/2}$ 380 Hz). [i] $\delta(^{13}C)$: 53.35 (NCH_2), 7.93(CH_2). [j] $\delta(^{13}C)$: 197.28(CS_2), 49.04(NCH_2), 11.83(CH_3).

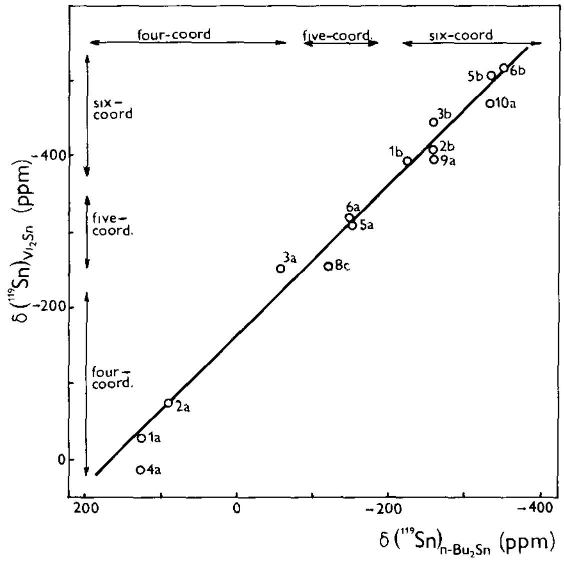

Fig. 1. Correlation of ^{119}Sn chemical shifts in divinyltin(IV) compounds with ^{119}Sn chemical shifts in di-n-butyltin(IV) analogues. Numbering of compounds as in Table 1 (a = $CDCl_3$, b = dmso-d_6, c = CD_3NO_2).

-220 ppm for four-coordinated, -250 to -350 ppm for five-coordinated and -375 to -520 ppm for six-coordinated tin atom in divinyltin(IV) compounds.

As expected the divinyltin(IV) halides and the compound $Vi_2Sn(SBu\text{-}n)_2$ in the solution of a non-coordinating solvent (deuteriochloroform) are present in the form of simple, almost isolated molecules with pseudotetrahedral coordination of the central tin atom. A relatively large up-field shift in $\delta(^{119}Sn)$ of divinyltin diiodide (compound **3**) in deuteriochloroform such that it infringes on the region typical of five-coordinated compounds is not exceptional. Similar "anomalous" shifts have been found for other diorganotin diiodides [5] which is probably due to partial double-bond character of the Sn=I connection, as postulated in ref. 8. We would like to add that the group of four-coordinate divinyltin(IV) compounds is sufficiently large and also includes several compounds containing at least two vinyl groups directly bonded to the tin atom and having various other substituents both polar and unpolar. The highest upfield shift value was that of Vi_4Sn (-160.3 ppm) [9]; the compounds $Vi_2SnR^1R^2$ ($R^1 = R^2 =$ Me, Et, n-Bu) give the values of $\delta(^{119}Sn)$ ranging from -79.9 to -86.4 ppm, and the trivinyltin(IV) compounds ($R^1 =$ Vi, $R^2 =$ Me, Et, Cl, Br, I) give $\delta(^{119}Sn)$ values between -44.7 and -124.0 ppm [9].

From the above estimation, the range of ~ -250 to -350 ppm can be said to be typical of five-coordinate divinyltin(IV) compounds. The studied divinyltin(IV) compounds were used to derive the values of $\delta(^{119}Sn)$ lying in this range for the complex, $Et_4N^+[Vi_2SnCl_3]^-$, and also for two divinyltin(IV) dicarboxylates **5** and **6**. We assume that the complex ion of $Vi_2SnCl_3^-$ probably has a *cis*-trigonal bipyramid arrangement of substituents around the central tin atom (vide infra), but the coordination number five of the tin atom in both carboxylates is only apparent. As a matter of fact the structure consists of six-coordinate asymmetric chelate with anisobidentate function of both carboxylate groups [10]. Such a structural model is supported by down-field shift of $\delta(^{13}C)$ of the carbon atom in the carboxylate group of this compound when non-coordinating deuteriochloroform solvent is replaced by a coordinating solvent (hexadeuteriodimethylsulphoxide) (vide infra) [11].

Divinyltin(IV) dioxinate and dithiocarbamate (compounds **9** and **10**) in deuteriochloroform, and each of the compounds in solutions of hexadeuteriodimethylsulphoxide reveal chemical shifts $\delta(^{119}Sn)$ characteristic of six-coordinate complexes. In the first two compounds it is due to the chelate function of bidentate ligands, in the other compounds the formation of six-coordinate complexes is explained by an equilibrium reaction with two molecules of coordinating solvents:

$$Vi_2SnX_2 + 2\text{dmso-}d_6 \rightleftharpoons Vi_2SnX_2 \cdot 2\text{dmso-}d_6 \qquad (2)$$

(X = Cl, Br, I, MeCOO, PhCOO, SBu-n and NCS)

The character of the substituent X affects both the reaction equilibrium (2) and the value of the chemical shift $\delta(^{119}Sn)$. In the case of Vi_2SnX_2 with X = Cl, Br, I and SBu-n the reaction of compound with solvent takes place as a simple addition of two solvent molecules via a donor–acceptor interaction of the donor oxygen atom of the solvent with the two vacant orbitals of the central tin atom. The formation of such complexes occurs readily in the case of divinyltin dihalides; the equilibrium here is shifted substantially to the right in favour of products and the measured $\delta(^{119}Sn)$ chemical shifts reveal mainly "natural" values of complexes [12]. A slight up-field shift of $\delta(^{119}Sn)$ for $Vi_2Sn(SBu\text{-}n)_2$ due to the replacement of deuteriochloroform (non-coordinating solvent) by hexadeuteriodimethylsulphoxide (coordinating solvent) is caused by the low tendency of organotin(IV) thiolates towards the formation of complexes. The equilibrium (2) is only slightly shifted to the right and thus $\delta(^{119}Sn)$ value of -24.4 ppm is only the weighted average of "natural" values of $\delta(^{119}Sn)$ for $Vi_2Sn(SBu\text{-}n)_2$ (which correspond to the value of the chemical shift determined in deuteriochloroform, $+16.3$ ppm) and for complexes of this compound with one or two molecules of solvent (these values are, however, unknown). The contribution of five- and six-coordinate complexes is very low. For the compounds with X = MeCOO or PhCOO, the equilibrium (2) is associated with the breakdown of the weak asymmetric chelate formed by the donor–acceptor bond between the oxygen atom of the carbonyl group and the central tin atom $C{=}O \cdots Sn$. In the complexes $Vi_2Sn(OCOR)_2 \cdot 2dmso\text{-}d_6$ (R = Me, Ph), the COO ligand is monodentate (vide infra). The compound $Vi_2Sn(NCS)_2$ in the solid state forms relatively strong polymers, with bridging NCS groups [13]. This arrangement results in a low solubility of the solid compound in non-polar (non-coordinating) solvents. In coordinating solvents breakdown of bridges and the formation of complexes with two solvents molecules takes place (the NCS group in this case is monodentate).

Coupling constants $^nJ(^{119}Sn, ^{13}C)$

The coupling constants $^1J(^{119}Sn, ^{13}C)$ of the studied compounds are listed in Table 1, the coupling constants $^2J(^{119}Sn, ^{13}C)$ are generally to small (< 15 Hz) to be detected.

The coupling constants $^1J(^{119}Sn, ^{13}C)$ of the studied divinyltin(IV) compounds and similar di-n-butyltin(IV) compounds [5] also reveal an excellent linear correlation which can be described by the equation:

$$^1J(^{119}Sn, ^{13}C)_{Vi_2Sn} = (1.65 \pm 0.08)\,^1J(^{119}Sn, ^{13}C)_{n\text{-}Bu_2Sn} - (1.07 \pm 57.43) \qquad (3)$$
$$N = 14, \ r = 0.984$$

We recently [14] described the correlation of the shapes of coordination polyhedra around central tin atom, i.e. the C–Sn–C bonds angles (θ angles), with the coupling constants $^1J(^{119}Sn, ^{13}C)$ of the n-butyltin(IV) compounds given by the equation

$$^1J(^{119}Sn, ^{13}C) = (9.99 \pm 0.73)\theta - (746 \pm 100) \qquad (4)$$

The linear character of the mutual correlation of the coupling constants $^1J(^{119}Sn, ^{13}C)$ given by eq. 3 justifies the supposition that a similar relation exists between $^1J(^{119}Sn, ^{13}C)$ and the θ angles for vinyltin(IV) compounds. A direct determination of such a relation is as yet impossible owing to insufficient data on the angles θ in vinyltin(IV) compounds.

On the basis of the correlation eqs. 3 and 4, and the assumption that there is not a substantial difference between the bond angles in corresponding divinyl- and dibutyltin(IV) compounds, the values of $^1J(^{119}Sn, ^{13}C)$ of divinyltin(IV) compounds lying in the range of ~ 550–750 Hz can be ascribed to the more or less deformed tetrahedral molecules of the simple Vi_2SnX_2 compounds (X = Cl, Br, I, SBu-n). The values of $^1J(^{119}Sn, ^{13}C)$ in the range of ~ 1450–1600 Hz can be ascribed to the *trans*-octahedral geometry of $Vi_2Sn_2 \cdot 2dmso\text{-}d_6$ (X = Cl, Br, I, NCS, MeCOO, PhCOO) complexes which is consistent with the structure deduced from the Mössbauer spectra [13]. The $^1J(^{119}Sn, ^{13}C)$ coupling constants which lie in the range

~ 900–1050 Hz should correspond to C–Sn–C angles of 135–145°. Thus the complex anion, $Vi_2SnCl_3^-$, is severely deformed *cis*-trigonal bipyramid, and the chelates of **5**, **6**, **9** and **10** are trapezoidal bipyramids.

Chemical shifts $\delta(^{13}C)$

The values of the chemical shifts $\delta(^{13}C)$ of both carbon atoms of the vinyl group in the studied compounds lie in a relatively broad range from 134.12 to 152.67 ppm for C(1) and 129.23 to 139.53 ppm for C(2). All the $\delta(^{13}C)$ values are shifted downfield in comparison with $\delta(^{13}C)$ of ethylene (123.3 ppm) [15]). There is a good mutual linear correlation between the $\delta(^{13}C)$ values of both carbon atoms in divinyltin(IV) compounds:

$$\delta(^{13}C)_{C(2)} = -(0.50 \pm 0.06)\delta(^{13}C)_{C(1)} + (205.65 \pm 8.03) \qquad (5)$$
$$N = 16, \ r = 0.922$$

The values of $\delta(^{13}C)$ for complexes **9** and **10** deviate slightly from this linear correlation. (The correlation parameters without taking into account those of **9** and **10** are: slope, 0.53 ± 0.03; intercept, 209.41 ± 4.54; $N + 14$, $r = 0.978$). Thus we can say that the change in the chemical shift in the $\delta(^{13}C)$ of carbon atom C(2) is accompanied by approximate halving of the $\delta(^{13}C)$ of C(1) in the opposite direction.

The values of $\delta(^{13}C)$ for both carbon atoms of vinyl groups in substituted ethylenes depend on several factors, some of which are contrary to each other, which makes analysis very difficult, especially in ethylenes substituted by an organometallic group, so the conclusions drawn from this analysis are only qualitative. Nevertheless, some relations are evident:

(i) Internal chemical shift [15] (and/or the polarization of double bond [16]) $\Delta\delta$ defined as the difference in the chemical shift ($\delta(^{13}C)$) of carbon atoms C(1) and C(2), i.e. $\Delta\delta = \delta(^{13}C)_{C(1)} - \delta(^{13}C)_{C(2)}$, for all of the tetrahedral divinyltin(IV) compounds under study is negative, whereas for the five- and six-coordinate complexes $\Delta\delta$ is positive. The electron density on the vinyl group in the first case shifts towards C(1), while in the latter case shifts towards C(2) atom [16]. This effect is probably due to the influence of the dipole of the organometallic substituents on the π-electron system of the vinyl group (π-polarization).

(ii) In vinyltin(IV) compounds the major influence of a substituent on the chemical shift of C(1) atom arises from the inductive effect [6,17]. This effect operates through the σ-bond and usually reveals dominant changes in σ-electron density on this carbon atom. Changes in π-electron density on this atom are substantially lower [6,17], but can play an important role, especially in the case of organometallic substituents. In agreement with this statement, the values of $\delta(^{13}C)$ of the C(1) atom of the compounds under study shift down-field with an increasing electronegativity of substituents (with increasing *s*-electron character of the Sn–C bond) [6]. This is manifested especially in the case of a change in coordination number of the central tin atom, e.g. for the series of compounds Vi_2SnCl_2, $Vi_2SnCl_3^-$ and $Vi_2SnCl_2 \cdot 2dmso\text{-}d_6$ with values of $\delta(^{13}C)$ for C(1) atom of 134.12, 148.53 and 152.52 ppm, respectively (the hybridisation changes from sp^3 via sp^2 to sp, respectively). But the steady increase in the $\delta(^{13}C)$ values of carbon atoms bonded directly to the tin atom, with increasing coordination number of the central tin atom, is a general effect dependent only to a small extent on the nature of substituents. This effect is supported also by almost the same differences in the values of $\delta(^{13}C)$ for atom C(1) between four-, five- and six-coordinated tri- and diorganotin(IV) compounds regardless of the character of organic substituent (n-butyl, fenyl, benzyl) as was observed earlier [1-3,5]. The influence of the inductive effect in compounds with the same coordination of the central tin atom is not as pronounced. We asume that the effect here is to a great extent counteracted by π-polarization (vide supra).

(iii) In contrast to the carbon atoms C(1), the σ-electron density on the C(2) carbon depends only slightly on the nature of substituents and the overall changes in the electron density on this atom is determined mainly by the contribution of π-electrons [16,17]. Consistent with the polarization of the C=C bond (corresponding to the sign of $\Delta\delta$) the values of $\delta(^{13}C)$ of the carbon atom C(2) in five- and six-coordinate divinyltin(IV) compounds are shifted up-field (129.23–132.44 ppm) compared with those for four-coordinate compounds (137.24–139.53 ppm). The value of $\delta(^{13}C)$ for C(2) of the complex $Vi_2SnCl_3^-$ is rather exceptional. Thus, the substituent of ethylene (i.e. the rest of molecules and complex ions of the studied compounds without a vinyl group) with a five- or six-coordinated central tin atom are stronger π-donors (or weaker π-acceptors) then the substituents with a four-coordinated tin atom [3].

(iv) The distinct differences in the values of $\delta(^{13}C)$ of both carbon atoms of the vinyl group, which are dependent on the coordination of central tin atom, can be also used (however on a smaller scale than in the case of $\delta(^{119}Sn)$) to determining the nature of the stereochemistry about the central tin atom in divinyltin(IV) compounds.

The values of $\delta(^{13}C)$ of carbon atoms in carboxylate groups of compounds **5** and **6** depend on the nature of the solvent. In agreement with the results of our recent

Table 2

Analytical data and melting (boiling) points of divinyltin(IV) compounds

Compound [a]	Analysis (Found (calc) (%))				m.p. (b.p.) (°C/Pa)
	C	H	Sn	Other	
3	10.98 (11.11)	1.28 (1.40)	27.84 (27.44)	I, 59.70 (60.06)	(128-130/100)
4	41.73 (41.05)	6.69 (6.89)	32.68 (33.80)	S, 18.51 (18.26)	(102-103/200)
5	32.43 (33.03)	3.42 (4.16)	41.25 (40.80)		260
6	52.30 (52.09)	3.69 (3.89)	28.38 (28.60)		154 (dec.)
8	36.03 (35.21)	6.45 (6.40)	28.69 (28.99)	N, 3.43 (3.42) Cl, 25.00 (25.98)	99-101
9	58.17 (57.31)	4.07 (3.93)	24.95 (25.74)	N, 6.45 (6.07)	159-160

[a] See Table 1.

papers [10,11] monodentate COO groups in complexes of divinyltin(IV) carboxylates **5** and **6** with two molecules of hexadeuteriodimethylsulphoxide have $\delta(^{13}C)_{COO}$ values shifted up-field (176.90 and 169.24 ppm, respectively) in comparison with anisobidentate carboxylate groups in assymmetric chelates **5** and **6** present in deuteriochloroform solutions (181.49 and 173.59 ppm, respectively).

Experimental

The compounds **1**, **2**, **7** and **10** were prepared by published procedures [13, 18 and 19], the other compounds were prepared by procedures described for analogous diorganotin(IV) compounds [20–22]. The results of elemental analysis of the compounds **3**–**6**, **8** and **9** together with melting and boiling points are listed in Table 2.

The ^{13}C, ^{14}N and ^{119}Sn NMR spectra were measured at 25.047, 7.195 and 37.14 MHz, respectively, on a JEOL spectrometer JNM-FX 100 at 300 K. The samples were as ca. 20% (w/v) or saturated (in the case of poor solubility) solutions. Chemical shifts $\delta(^{119}Sn)$ are related to external neat tetramethylstannane, $\delta(^{14}N)$ to external neat nitromethane and $\delta(^{13}C)$ to the corresponding solvent signal and converted to the δ-scale: deuteriochloroform (77.00 ppm), trideuterionitromethane (62.60 ppm) and hexadeuteriodimethyl sulphoxide (39.60 ppm). Positive values denote down-field shift. A more detailed description of the experimental conditions is to be found in refs. 1, 2.

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