The Structure of Tetrachlorostannate Complexes with Ketones from ^{35}Cl NQR Data

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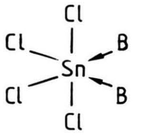
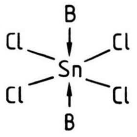
^{35}Cl NQR spectra (77 K) of 1:1 and 2:1 $RR'CO-SnCl_4$ are recorded and discussed. These spectra change in the course of time. At low temperatures either octahedral or trigonal-bipyramidal complexes (depending on the ratio of components and the nature of ligands) are formed. Frequently these systems contain a mixture of steric isomers or a mixture of complexes having different coordination numbers of the tin atom. In the solid state some complexes show a transition from one to another configuration.

Introduction

The $SnCl_4$-ketone complexes have not been studied much by ^{35}Cl NQR spectroscopy. It has only been reported that the $SnCl_4$ complex with acetone displays a cis-structure (I) [1] whereas that with acetophenone displays a trans-octahedral configuration (II) [2]. For the 1:2 $SnCl_4$-cyclohexanone complex a triplet ^{35}Cl NQR spectrum has been recorded, which is of no help in elucidating the cis- or trans-configuration of this complex [1]. We have extended the investigation of the tetrachlorotin-ketone complexes by ^{35}Cl NQR spectroscopy. The frequencies at 77 K are recorded in Table 1. We also traced the changes appearing in the $RR'CO-SnCl_4$ systems in the solid state in the course of time.

^{35}Cl NQR spectroscopy provides an effective method for studying these systems. It allows a fast and quite reliable characterization of the complexes, i.e. their structure, peculiarities of the electron distribution etc. (see [1–16]). We have shown that $SnCl_4$ complexes with organic compounds may be octahedral (I, II) and trigonal-bipyramidal (III), (see [3–6, 8, 11–15]). (I, II) and (III) are quite different.


I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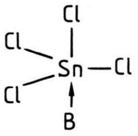
III

The ^{35}Cl NQR spectra of octahedral complexes consist of one ore more lines. The number of lines depends on the symmetry elements of the Sn atom coordination polyhedron and the number of unequal molecules in the crystal unit cell. All these lines usually appear at lower frequencies than those of $SnCl_4$. ^{35}Cl NQR frequencies of complexes studied are lower than 22.0 MHz. Trigonal-bipyramidal $SnCl_4$ complexes show two or more ^{35}Cl NQR lines, the number being determined by the same factors as for octahedral complexes. The lines of the equatorial Cl atoms of trigonal-bipyramidal $SnCl_4$ complexes are shifted slightly from those of $SnCl_4$, whereas the frequency of the line (or lines) of the axial Cl atom is considerably lower. The frequency difference between the axial and the equatorial Cl atoms is usually greater than 1.0 MHz. The identification of the structural types by means of NQR spectra conforms with X-ray data [5, 6] and ^{35}Cl nuclei EFG asymmetry parameters [13–15].

Results and Discussion

The ^{35}Cl NQR spectrum of 2:1 $(CH_3)_2CO-SnCl_4$ (prepared immediately prior to its measurement shows four lines with nearly equal intensity. The frequencies are considerably lower than those of $SnCl_4$. The spectrum is characteristic for octahedral $2\,B \cdot SnCl_4$ complexes [1–16]. The quadruplet inner lines are overlap-

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^{35}Cl frequencies at 77 K (ν^{77}) of trigonal-bipyramidal ($n=1$) and octahedral ($n=2$) nB · $SnCl_4$ complexes and signal/noise ratios (in parentheses),

B	ν^{77}, MHz			
	$n=1$		$n=2$	
1	2		3	
$(CH_3)_2CO$	–		20.120 (7)	19.490 (7)
			19.436 (7)	18.821 (6)
after 35 h	–		20.130 (28)	18.832 (11)
			18.778 (11)	
$CH_3C(O)C_2H_5$	–		20.276 (7)	19.508 (6)
			19.250 (6)	18.515 (4)
n-$C_3H_7C(O)CH_3$	22.997 (14)	22.856 (12)		
	22.521 (10)	19.035 (10)		
cryst. fast			19.947 (13)	19.554 (14)
			18.896 (12)	18.354 (13)
cryst. slowly			19.854 (13)	19.732 (18)
			19.212 (17)	19.095 (20)
kept. at 273 K			19.922 (16)	19.825 (16)
			19.135 (14)	18.824 (20)
$CH_3C(O)CH(CH_3)_2$	23.117 (18)	22.780 (18)	20.032 (3)	19.868 (3)
	22.708 (24)	19.626 (10)	19.155 (3)	18.922 (3)
$CH_3C(O)C(CH_3)_3$	23.554 (40)	22.766 (45)	20.497 (6)	20.381 (6)
	22.615 (40)	19.528 (25)	20.291 (7)	19.186 (4)
			19.040 (4)	18.909 (6)
			18.750 (6)	
$CH_3C(O)C_6H_5$	23.010 (55)	22.588 (25)	19.587 (4)	19.282 (2)
	22.387 (25)	19.640 (30)	19.081 (2)	
4-$CH_3C_6H_4C(O)CH_3$	22.977 (29)	22.890 (31)	19.909 (5)	19.752 (4)
	22.742 (6)	22.392 (6)	18.928 (4)	18.777 (5)
	22.145 (28)	19.460 (8)		
	19.139 (31)			
4-$ClC_6H_4C(O)CH_3$*	34.917 (50)	23.482 (22)	34.918 (25)	19.369 (4)
	23.040 (26)	22.297 (16)	19.234 (5)	
	19.356 (10)			
$(C_2H_5)_2CO$			20.160 (3)	20.015 (3)
			19.864 (5)	18.933 (2)
			18.663 (3)	18.600 (3)
			18.445 (4)	
$\overline{CH_2(CH_2)_3CO}$	22.950 (38)	22.888 (42)	–	
	22.793 (35)	20.100 (30)		
$\overline{CH_2(CH_2)_4CO}$	23.298 (30)	22.752 (28)	21.676 (8)	20.281 (7)
	22.390 (30)	19.595 (20)	18.910 (5)	17.947 (4)
$(C_6H_5)_2CO$	23.178 (30)	22.837 (22)	20.019 (8)	18.918 (6)
(components ratio 1:1)	19.533 (14)			
after 4 months	23.758 (18)	23.126 (26)		
	22.982 (20)	22.898 (21)		
	19.623 (20)			
$(C_6H_5)_2CO$	–		19.90 (6)	19.75 (6)
(components ratio 2:1)			19.38 (2)	

* For 4-$ClC_6H_4C(O)CH_3$, $\nu^{77}=34.620$ MHz, S/N = 60.

ping. With progressing time these lines fade whereas the highest frequency line strenghtens, the lowest frequency line being split. When the system is kept for 35 h at 313 K, its spectrum acquires a triplet character, resembling in frequencies the outer lines of the originally recorded spectrum. Two inner lines of the latter are absent in the triplet. The same ^{35}Cl NQR spectrum can be obtained when 2:1 $(CH_3)_2CO$–$SnCl_4$ is kept for a much longer time at room temperature. Judging from the ^{35}Cl NQR spectra, the system contains, immediately after preparation, a mixture of isomers of octahedral $2(CH_3)_2CO \cdot SnCl_4$. One of the isomers gives rise to the quadruplet outer lines, the other one to the inner lines. In each isomer two Cl atoms are pairwise equivalent. With progressing time, the second isomer becomes the first one in which the two Cl atoms show the same electron distribution. The highest-frequency line of the triplet corresponds to these Cl-atoms. If the system displaying the triplet is fused and then allowed to crystallize again, the spectrum acquires its original quadruplet appearance.

Previously, the transition of solid cis-octahedral 2 B · $SnCl_4$ complexes (B = N,N-dimethylacetamide, N,N-dimethylformamide and dimethylsulfoxide) to a trans-isomer (II) has been reported [17]. A more radical change was observed in the tin coordination polyhedron in the solid 1:1 $SnCl_4$ complex with ortho-methoxybenzoylchloride, i.e., a transition from the trigonal-bipyramidal to the octahedral structure [4]. The transition observed by us of the octahedral $2(CH_3)_2CO \cdot SnCl_4$ to the other steric isomer supplements the rather poor information available concerning the transformations of complexes in the solid state.

The ^{35}Cl NQR spectrum of 1:1 $(CH_3)_2CO$–$SnCl_4$ consists of the quadruplet spectrum of the $2(CH_3)_2CO \cdot SnCl_4$ complex and the spectrum of $SnCl_4$.

The NQR spectrum of 2:1 $CH_3C(=)C_2H_5$–$SnCl_4$ indicates the formation of an octahedral complex. The spectrum is analogous so that of the freshly prepared 2:1 $(CH_3)_2CO$–$SnCl_4$, assignable to two steric isomers of the octahedral complex. In contrast to the latter, however, the NQR spectrum of 2:1 $CH_3C(O)C_2H_5$–$SnCl_4$ does not change much with time and upon heating the system. In this case the lines fade and disappear when the system is kept at 313 K for over 5 days. These lines are also present in the NQR spectrum of 1:1 $CH_3C(O)C_2H_5$–$SnCl_4$, but they overlap with the ^{37}Cl NQR lines of the included $SnCl_4$ (19.137, 19.100, 19.030 and 18.689 MHz). The quadruplet ^{35}Cl NQR spectrum of the latter occupies the highest-frequency region of the NQR spectrum for 1:1 $CH_3C(O)C_2H_5$–$SnCl_4$ (Table 1).

For 2:1 *n*-$C_3H_7C(O)CH_3$–$SnCl_4$ one of the quadruplet ^{35}Cl NQR spectra is observed depending upon the crystallization rate of the system. The spectra indicate the formation of various steric isomers of $(n\text{-}C_3H_7C(O)CH_3)_2 \cdot SnCl_4$. It is also possible to obtain a mixture of these isomers, an eight-component NQR spectrum which consists of the quadruplet spectra of the isomers. When the quickly crystallizing system is kept at 273 K for about 2 days, its NQR spectrum is transformed to that of the slowly crystallizing system. When the latter is kept at the same temperature for about a day, the lines in its quadruplet NQR spectrum are shifted, remaining, however, in the same frequency range. This spectrum also belongs to the octahedral complex. The ^{35}Cl NQR spectrum of 1:1 $n\text{-}C_3H_7C(O)CH_3 \cdot SnCl_4$ contains the spectra of both $SnCl_4$ (4 highest frequency lines) and trigonal-bipyramidal $n\text{-}CH_3C(O)C_3H_7 \cdot SnCl_4$ (5 low-frequency lines, one of which is substantially shifted toward lower frequency.

The ^{35}Cl NQR spectrum of $(CH_3C(O)C_6H_5)_2 \cdot SnCl_4$ recorded by us (Table 1) nearly coincides with that reported previously [2] and confirms the octahedral structure of this complex. If $SnCl_4$ is added up to a 1:1 ratio of components, the octahedral complex is transformed to a trigonal-bipyramidal one. Its quadruplet NQR spectrum shows one line at a considerably lower-frequency than the other which appear at slightly lower frequencies than those of $SnCl_4$.

Judging from the ^{35}Cl NQR spectra (Table 1), with an equimolar ratio of B and $SnCl_4$ (B = $CH_3C(O)CH(CH_3)_2$, $CH_3C(O)C(CH_3)_3$ and $4\text{-}ClC_6H_4C(O)CH_3$ trigonal-bipyramidal complexes are formed, whereas octahedral complexes appear for the 2:1 ratio. The NQR frequency of Cl attached to the aromatic ring and, consequently, the electron distribution of this atom in octahedral $(4\text{-}ClC_6H_4C(O)CH_3)_2 \cdot SnCl_4$ coincide with the NQR frequency and electron distribution of the corresponding Cl in trigonal-bipyramidal $4\text{-}ClC_6H_4C(O)CH_3 \cdot SnCl_4$.

The NQR spectrum of the $(CH_3C(O)C(CH_3)_3)_2 \cdot SnCl_4$ complex contains 7 lines of various intensities. The spectrum may correspond either to several molecules of the complex in the crystal unit cell or to some isomers of the octahedral complex. The former is less probable since the line intensity ratio in the NQR spectrum is slightly dependent on the crystallization rate of the system. The possible presence of several steric isomers in the RR′CO–$SnCl_4$ systems is supported by the NQR spectrum of 1:1 $4\text{-}ClC_6H_4C(CO)CH_3\text{–}SnCl_4$. It contains two low-frequency and a group of high-frequency lines, all showing quite different intensities. The NQR frequencies of the high-frequency lines are markedly lower than those of $SnCl_4$ and occur in a range typical of the equatorial Cl atom in trigonal-bipyramidal RR′CO · $SnCl_4$ complexes (Table 1). Two low-frequency lines correspond to the axial Cl atoms in such complexes. This NQR spectrum unambiguously shows that 1:1 $4\text{-}CH_3C_6H_4C(CO)CH_3\text{–}SnCl_4$ includes two isomers of the trigonal-bipyramidal complex with a slightly different electron distribution of the corresponding Cl atoms. One of the isomers causes the 4 most intense lines, the other gives rise to three considerably weaker lines, the fourth one possibly overlapping one of the three most intense high-frequency lines. On the basis of the ratio of the most and the least intense lines in the NQR spectrum of 1:1 $4\text{-}CH_3C_6H_4C(O)CH_3\text{–}SnCl_4$, one of the two complexes is suggested to be 4 times more frequent than the other. After some time, this spectrum is transformed to a quadruplet. If the system is fused and then allowed to crystallize, its quadruplet spectrum does not change much. A similar spectrum can also be obtained for a freshly prepared system. The spectral appearance and the composition of the system in the solid state is likely to depend on its crystallization rate.

The quadruplet NQR spectrum of 2:1 $4\text{-}CH_3C_6H_4C(O)CH_3\text{–}SnCl_4$, consisting of two doublets fairly far apart from each other, indicates the formation of octahedral $(4\text{-}CH_3C_6H_4CH_3)_2 \cdot SnCl_4$. This spectrum does not change much when the system is kept for 5 days at 313 K.

The ^{35}Cl NQR spectrum of 2:1 $(C_2H_5)_2CO\text{–}SnCl_4$ suggests either to contain two different molecules of octahedral $((C_2H_5)_2CO)_2 \cdot SnCl_4$ in the crystal unit cell or to consist of an equimolar mixture of steric isomers of this type of complex. In the NQR spectrum of the 1:1 system some intense lines from $SnCl_4$ and weak lines from the 2:1 system were observed. The latter were overlapped by the ^{37}Cl NQR spectrum of $SnCl_4$.

The ^{35}Cl NQR spectrum of 2:1 $\overline{CH_2(CH_2)_4CO}\text{–}SnCl_4$ differs considerably from the triplet spectrum previously reported [1], two lines of which occur in a high-frequency range not common for octahedral $SnCl_4$ complexes. Our quadruplet NQR frequencies of this complex are in the same frequency range as the other octahedral 2B · $SnCl_4$ complexes (see, for example, [2, 7–10] and Table 1). Their frequencies are much lower than those of $SnCl_4$. The frequency range of the quadruplet appearing exceeds greatly the range attributable to the crystalline effect [16]. The low-frequency lines in the spectrum are slightly wider and less intense than the high-frequency lines. This spectrum seems to correspond to the $(\overline{\mathrm{CH_2(CH_2)_4CO}})_2 \cdot \mathrm{SnCl_4}$ complex showing a somewhat distorted octahedral structure. It is also possible that 2:1 $\overline{\mathrm{CH_2(CH_2)_4CO}}$–$\mathrm{SnCl_4}$ contains a mixture of two steric isomers of the octahedra complex.

The ^{35}Cl NQR of the 1:1 $\overline{\mathrm{CH_2(CH_2)_4CO}}$–$\mathrm{SnCl_4}$ shows also a quadruplet. The frequencies of its high-frequency lines are somewhat lower than those $\mathrm{SnCl_4}$, whereas the lowest-frequency line is considerably remote from these. This spectrum corresponds to trigonal-bipyramidal $\overline{\mathrm{CH_2(CH_2)_4CO}} \cdot \mathrm{SnCl_4}$. The NQR frequencies of one low-frequency and three high-frequency lines in this spectrum are close to those observed previously in the triplet spectrum.

Consequently, the latter, assigned to octahedral $2\,\overline{\mathrm{CH_2(CH_2)_4CO}} \cdot \mathrm{SnCl_4}$ is, in fact, an incomplete spectrum of trigonal-bipyramidal $\overline{\mathrm{CH_2(CH_2)_4CO}} \cdot \mathrm{SnCl_4}$. In the bipyramid equatorial position there are three Cl atoms giving rise to three high-frequency lines, whereas in the axial position there is a fourth Cl atom. The latter causes the low-frequency line in the spectrum (Table 1).

The ^{35}Cl NQR spectrum of 1:1 $\overline{\mathrm{CH_2(CH_2)_3CO}}$–$\mathrm{SnCl_4}$ is analogous to that of trigonal-bipyramidal $\overline{\mathrm{CH_2(CH_2)_4CO}} \cdot \mathrm{SnCl_4}$. This indicates that the system contains a complex of the same structure. It was not possible to obtain the NQR spectrum of this system with 2:1 composition.

2:1 $\mathrm{(C_6H_5)_2CO}$–$\mathrm{SnCl_4}$ shows some piezoelectric properties. Its triplet spectrum recorded without suppressing these properties points to the formation of octahedral $2\mathrm{(C_6H_5)_2CO} \cdot \mathrm{SnCl_4}$. It is most likely that only the most intense spectral lines of this system can be detected by this procedure. Upon complete suppression of piezoelectric properties it was not possible to observe the spectrum of this system.

The ^{35}Cl NQR spectrum of the equimolar mixture of $\mathrm{(C_6H_5)_2CO}$ and $\mathrm{SnCl_4}$ consists of the spectrum of $\mathrm{SnCl_4}$ and those of structurally different complexes of $\mathrm{SnCl_4}$ with $\mathrm{(C_6H_5)_2CO}$. The most intense line in the spectrum (23.178 MHz) is significantly wider (its half-height width, $\Delta\nu \approx 0.13$ MHz) than the neighbouring one (22.837 MHz, $\Delta\nu \approx 0.09$ MHz). This seems to arise from two Cl atoms. The frequencies of these two lines are much lower than those of $\mathrm{SnCl_4}$. They are typical of the equatorial chlorines in trigonal-bipyramidal $\mathrm{SnCl_4}$ complexes (see Table 1). The axial chlorine of this complex evidently gives rise to a line with the NQR frequency (19.533 MHz) close to those of analogous atoms in trigonal-bipyramidal complexes, such as $\mathrm{B \cdot SnCl_4}$, for example, where $\mathrm{B = CH_3C(O)C_6H_5}$, $\overline{\mathrm{CH_2(CH_2)_3CO}}$ and $\overline{\mathrm{CH_2(CH_2)_4CO}}$. The two remaining lines in the spectrum (20.019 and 18.918 MHz) are due to the octahedral complex $2\mathrm{(C_6H_5)_2CO} \cdot \mathrm{SnCl_4}$. Each of the lines corresponds to two Cl atoms. Nevertheless, these lines are noticeable weaker than those for the axial Cl atom in the trigonal-bipyramidal $\mathrm{(C_6H_5)_2CO} \cdot \mathrm{SnCl_4}$ complex. This suggests that the octahedral complex in 1:1 $\mathrm{(C_6H_5)_2CO}$–$\mathrm{SnCl_4}$ is several times less frequent than the trigonal-bipyramidal one. The amount of the octahedral complex and $\mathrm{SnCl_4}$ in the solid system decreases depending on the elapsing time and the temperature of the system. In this case the corresponding lines in the NQR spectrum fade and the highest-frequency line belonging to the trigonal-bipyramidal complex (23.212 and 23.173 MHz) is split. When the system is kept for about four months at room temperature, its NQR spectrum becomes a five-component one (Table 1). The lowest-frequency line in this spectrum is far apart from the high-frequency quadruplet observed in a markedly lower-frequency range than the spectrum of $\mathrm{SnCl_4}$. One of the quadruplet lines (23.126 MHz) is much more intense and wider than other lines. Its width is $\Delta\nu \approx 0.12$ MHz compared with $\Delta\nu \approx 0.07$ MHz for other quadruplet lines. This spectrum appears to correspond to a mixture of two isomers of the trigonal-bipyramidal complex, exhibiting the same electron distribution of axial Cl atoms. These atoms give rise to the lowest-frequency line in the spectrum. The electron distribution of the equatorial Cl atoms in these complexes is slightly different. Thus, the solid mixture of trigonal-bipyramidal and octahedral complexes is transformed, in the course of time, to a mixture of two isomers of the trigonal-bipyramidal complexes.

Conclusion

The data obtained provide evidence that at low temperature the complexes of $\mathrm{SnCl_4}$ with ketones may be octahedral, but also trigonal-bipyramidal, depending on the ratio of the components and the nature of ligand. The common feature of all the trigonal-bipyramidal complexes of $\mathrm{SnCl_4}$ with ketones RR′CO (as well as those with esters RCOOR′ [8]) is a substantial change (compared with pure $SnCl_4$) in the electron distribution in not only the axial, but also in the equatorial Cl atoms. Their NQR frequencies are considerably lower than those observed for $SnCl_4$. In these complexes, the electron density of the axial chlorine and, consequently, the partial negative charge on it, are noticeably higher than in the $SnCl_4$ complexes with chloroanhydrides of carboxylic acids (see, for example, [3, 11]) or with ethers [12].

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