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COMPLEXES OF TETRAALKYLALUMINOXANES WITH SOME NITROGEN-CONTAINING LEWIS BASES

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Summary

Tetraalkylaluminoxanes, R_4Al_2O (where R = Et, i-Bu), were found to form donor—acceptor complexes with some nitrogen containing electron donors (NMe_2Ph, PhHC=N-n-Bu and PhCN). These complexes are generally monomeric and have a 1/1 molar ratio of the components. A structure, based on low temperature PMR spectral studies, is postulated for each complex with the bridging nitrogen atom bonded to both aluminium atoms in the complexed aluminoxane. Some steric effects during the complex formation are discussed. It is shown that they are considerably responsible for the complexation.

Introduction

The unusual electron-accepting nature of the R_4Al_2O compounds, due to the bidentate property of two aluminium atoms in a molecule has already been observed [1—6]. Compounds of the type R_4Al_2O (where R = Me, Et, i-Bu, octyl and Cl) were found to form stable, mono- or di-meric complexes with electron donors, e.g. diethyl ether [1—3], trimethylamine [4], tetrahydrofuran, anisole or 5,6-benzoquinoline (BQ) [5]. In general, the molar ratio of the components in these complexes is 1/1. Complexes containing more than one donor molecule per one Al—O—Al linkage, e.g. $Et_4Al_2O \cdot 2$ BQ, are less stable and undergo dissociation to give the free donor and the corresponding monocoordinated complex [5]. However, for some donor molecules which have an easier steric access to aluminium atoms in the aluminoxane, the formation of stable $R_4Al_2O \cdot m$Do complexes ($m \leqslant 2$, Do = donor molecule) was found [6]. This was observed for benzonitrile complexes with chloromethylaluminoxanes of the general formula $Me_nCl_{4-n}Al_2O$ for $n = 0, 2$. This phenomenon is additionally due to the increasing acidity of the aluminoxane, caused by the replacement of the aluminium methyl group by chlorine. A small steric effect and the increased acidity of aluminium atoms in the case of chloromethylaluminoxanes is also responsible for the association of the benzonitrile [6] and diethyl ether complexes [2,3]. For more bulky groups attached to aluminium the association of the complexes does not occur [1].

Recently [7] we have described the tetraethylaluminoxane—benzonitrile complex. Structure I of this complex was proposed as more probable than the corresponding dynamic one. Such a proposal is in agreement with the bidentate electron-acceptor nature of the compound ("chelaptor") [5]. The present study concerns the formation and the structure of complexes of R_4Al_2O with other nitrogen-containing Lewis bases.

Results and discussion

Addition of a Lewis base to the aluminoxane solution leads to complex formation according to eqn. 1

$$[R_4Al_2O]_n + n\,\mathrm{Do} \rightarrow nR_4Al_2O \cdot \mathrm{Do} \qquad (1)$$

$(n = 2,3)$

The complexes obtained were characterized by PMR and IR spectral studies and cryoscopic molecular weight determinations.

The proton magnetic resonance spectra of the investigated complexes show several interesting features. Tables 1 and 2 list the observed chemical shifts of the aluminoxane alkyl group protons and the characteristic protons of a donor molecule. Firstly, in the case of tetraethylaluminoxane complexes (Table 1), the signals attributable to the bridging ethyl groups in the aluminoxane do not occur [7,8]. The lone electron pair of the donor replaces the bridging alkyl group. Secondly, the signals of methylene group protons (—CH_2Al) shift downfield in the case of the benzonitrile complex (PhCN, —0.17 ppm) whereas the complex formation with other amines shifts them upfield: *N*,*N*-dimethylaniline (NMe_2Ph, +0.22 ppm) and benzylidene-n-butylamine (PhHC=N-n-Bu, SB, +0.30 ppm). Also, the internal chemical shifts of the aluminoxane ethyl group increases. For tetraisobutylaluminoxane complexes similar changes in the chemical shifts of CH_2Al protons are also observed (Table 2).

The observed changes are characteristic for the complex formation between aluminoxanes and the investigated donors. They are in the direction expected on electronic grounds, as the coordinate link supplies electronic charge to the aluminium with consequent electron drift to the aluminium-attached alkyl groups. Moreover, the magnitudes of these changes may afford some evidences of the relative donor properties of the investigated donors.

The usual manner to describe the strength of a base, in terms of the dissociation constant K_a of its conjugate positive ion, i.e. the constant of reaction 2, gives the values of pK_a —10.4 for benzonitrile [9], 5.12 for *N*,*N*-dimethylaniline

$$BH^+ + H_2O \rightleftharpoons B + H_3O^+ \qquad (2)$$

[10] and 6.7 for benzylidene-n-butylamine [11]. The stronger the base the higher is the pK_a value. The observed changes in the chemical shifts of the methylene group protons attached to the aluminium, as well as the changes in the internal chemical shifts of the aluminoxane ethyl group, correlate well with

TABLE 1

THE COMPARISON OF PROTON MAGNETIC RESONANCE SPECTRA OF TETRAETHYLALUMINOXANE COMPLEX WITH THE CORRESPONDING TRIETHYLALUMINIUM ANALOGUES

(Solvents: toluene, benzene and methylene chloride as internal standards at τ 7.66 ppm, τ 2.72 ppm, τ 4.70 ppm, respectively)

Compound	Temperature (°C)	Solvent	PMR resonances			
			Organoaluminium compounds			Donor
			CH_3 (t) (ppm)	CH_2 (q) (ppm)	δ (Et) (ppm)	(ppm)
Et_3Al [8]	+25	toluene	8.70	9.48	0.78	—
	−70		8.51 [a]	9.61 [a]	1.10	—
			8.84 [b]	9.11 [b]	0.27	—
Et_4Al_2O [8]	+25		8.66	9.46	0.80	—
	−70		8.56 [a]	9.59 [a]	1.03	—
			8.87 [b]	9.08 [b]	0.21	—
PhCN	+25	methylene chloride				2.48 [c]
	−70					2.52
Et_3Al + PhCN, 1/1	+25	toluene	8.27	9.43	1.16	
	−70		8.06	9.16	1.10	
Et_3Al + PhCN, 1/1	+25	methylene chloride	8.90	10.12	1.22	2.27
	−70		8.97	10.22	1.25	2.30
Et_3Al + PhCN, 1/2	+25		8.90	10.12	1.22	2.39
	−70		8.98	10.22	1.24	2.41
Et_4Al_2O + PhCN, 1/1	+25	toluene	8.27	9.42	1.15	
	−70		8.03	9.13	1.10	
Et_4Al_2O + PhCN, 1/2	+25		8.27	9.42	1.15	
	−70		8.10	9.21	1.11	
Et_4Al_2O + PhCN, 1/1	+25	methylene chloride	8.95	10.18	1.23	2.30
	−70		9.09	10.25	1.16	2.30
Et_4Al_2O + PhCN, 1/2	+25		8.95	10.18	1.23	2.40
	−70		9.06	10.25	1.19	2.40
NMe_2Ph	+25	toluene				7.19 [d]
	−70					7.31
Et_3Al + NMe_2Ph, 1/1	+25		8.50	9.74	1.24	7.39
	−70		8.32	9.58	1.26	7.46
Et_3Al + NMe_2Ph, 1/1	+25		8.52	9.76	1.24	7.30
	−70		8.29	9.57	1.28	7.38
Et_4Al_2O + NMe_2Ph, 1/1	+25		8.58	9.81	1.23	7.34
	−70		8.41	9.69	1.28	7.34
Et_4Al_2O + NMe_2Ph, 1/2	+25		8.57	9.82	1.25	7.26
	−70		8.40	9.69	1.29	7.26
PhHC=N-n-Bu (SB)	+25	toluene				6.30 [e]
	−70					6.30
Et_4Al_2O + SB, 1/1	+25		8.47	9.89	1.42	6.35
	−70		8.24	9.41	1.17	6.30 (br)
Et_4Al_2O + SB, 1/2	+25		8.50	9.91	1.41	6.32
	−70		8.24	9.41	1.17	6.31 (br)

[a] Terminal. [b] Bridging. [c] The average chemical shift of ring protons of Ph group in PhCN, multiplet. [d] The chemical shift of methyl group protons in NMe_2Ph, singlet. [e] The chemical shift of methylene $-CH_2-N=$ group protons in SB, triplet.

TABLE 2

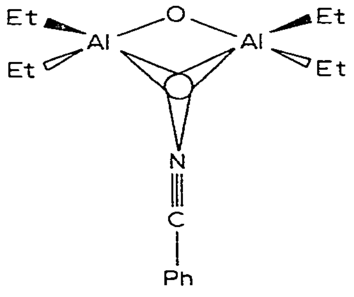
PROTON MAGNETIC RESONANCE SPECTRA OF TETRAISOBUTYLALUMINOXANE COMPLEXES
(Solvents: toluene and benzene as internal standards at τ 7.66 ppm and τ 2.72 ppm, respectively. Temperature +25°C)

Compound	Solvent	PMR resonances			
		Organoaluminium compounds			Donor
		CH_3 (ppm)	CH_2 (ppm)	δ(i-Bu) (ppm)	(ppm)
i-Bu_4Al_2O [24]	toluene	8.63 (d) 8.56 (br)	9.51 (d) 9.31 (br)	0.88	
i-Bu_4Al_2O [24]	benzene	8.70 (d) 8.63 (br)	9.62 (d) 9.41 (br)	0.88	
i-Bu_4Al_2O + PhCN	toluene	8.49 (d) 8.44 (br)	9.33 (d) 9.21 (br)	0.84	
NMe_2Ph	benzene				7.30 [a]
i-Bu_4Al_2O + NMe_2Ph	benzene	8.72 (d) 8.65 (br)	9.61 (d) 9.41 (br)	0.89	7.31

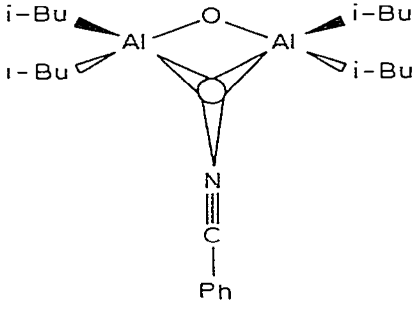
[a] The chemical shift of NMe_2Ph methyl groups protons, singlet.

the order of the calculated pK_a values. The order of basicity strength for the investigated donors can be written as follows: PhCN << NMe_2Ph < PhHC=N-n-Bu. But their donor properties toward aluminoxanes may be influenced by the electronic and steric effects acting on the functional nitrogen atom.

The nitrile group appears to be a much weaker donor than the nitrogen atom in the investigated amines. Due to its linear C—C≡N arrangement, benzonitrile will, as a rule, not be subject to steric hindrance regarding complexation. However, when the acceptor is strongly sterically screened, steric factors prevent the close proximity of the donor and the acceptor necessary for the formation of a stable complex. The tetraethylaluminoxane—benzonitrile complex (I) is stable, monomeric and of a 1 : 1 stoichiometry as was found previously [7]. The nitrogen atom of the nitrile group has in this case an easy steric access to the aluminoxane aluminium atoms. The tetraisobutylaluminoxane—benzonitrile complex (II) is found by IR spectrum (Table 3) and cryoscopic molecular weight determinations (Table 4) to be in equilibrium with the free substrates (eqn. 3). In the



(I)



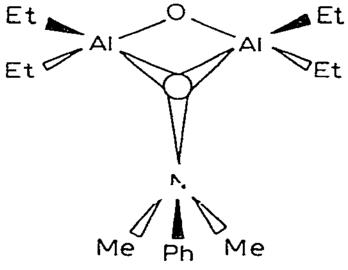
(II)

$$\text{i-Bu}_4\text{Al}_2\text{O} \cdot \text{PhCN} \rightleftharpoons \text{i-Bu}_4\text{Al}_2\text{O} + \text{PhCN} \qquad (3)$$

(II)

IR spectrum of a 1/1 mixture of tetraisobutylaluminoxane with benzonitrile, in benzene, a weak band at 2230 cm^{-1} attributable to uncomplexed benzonitrile appears. The measured molecular weight of this mixture is the average of the complex and free substrates.

In contrast, the tetraethylaluminoxane—*N*,*N*-dimethylaniline complex (III) is also an equilibrium one (eqn. 4), although NMe_2Ph is a much stronger base than benzonitrile. This equilibrium is strongly shifted to the complexation, however, a very weak signal due to the uncomplexed amine appears in the IR spectrum of its equimolar mixture with $\text{Et}_4\text{Al}_2\text{O}$ at 1059 cm^{-1} [12] (Table 3). The molecular weight determinations confirm the equilibrium state of the complex (Table 4).



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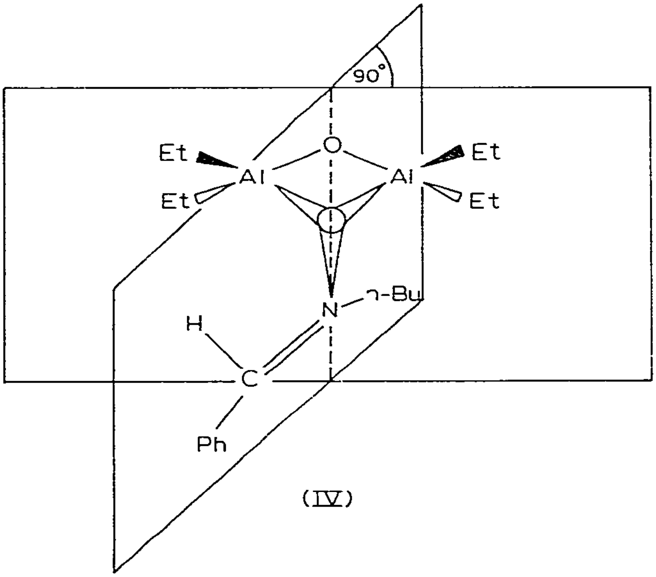


(IV)

$$\text{Et}_4\text{Al}_2\text{O} \cdot \text{NMe}_2\text{Ph} \rightleftharpoons \text{Et}_4\text{Al}_2\text{O} + \text{NMe}_2\text{Ph} \qquad (4)$$

(III)

Tetraisobutylaluminoxane does not form a complex with NMe_2Ph at all. IR and PMR spectral results as well as molecular weight determinations (Tables 2—4) confirm this conclusion.

The difficulties observed during the complexation by *N*,*N*-dimethylaniline agree well with its molecular structure, where the nitrogen atom is considerably sterically screened. There is a discrepancy in the literature [10] concerning the geometry of aromatic amines; it seems possible that in the liquid phase the nitrogen atom and both methyl and phenyl groups do not lie in one plane, though the crystal structure of a similar arrangement is evidently planar [10]. The most probable structure of the complexed NMe_2Ph seems to be the trigonal pyramid with the lone electron pair directed along the main axis of the molecule.

TABLE 3

IR SPECTRA OF INVESTIGATED COMPLEXES IN THE RANGE 2300—600 cm^{-1}

Compound	Chracteristic donor vibration (cm^{-1})	Free donor (cm^{-1})	$\Delta\nu$ (cm^{-1})	Assignment
Et_4Al_2O + PhCN	2270 (vs)	2230 (vs)	+40	$\nu(C\equiv N)$
i-Bu_4Al_2O + PhCN	2269 (vs)		+39	
	2230 (w)			
Et_4Al_2O + NMe_2Ph	1011 (m)	1059 (s)	—48	$\nu_{as}(-\overset{\vert}{\underset{\vert}{C}}-N)$
	1059 (w)			
	1344 (s)	1344 (s)	0	$\nu_{as}(\gtrdot C-N)$
i-Bu_4Al_2O + NMe_2Ph	1059 (s)		0	
	1034 (s)		0	
Et_4Al_2O + SB	1628 (vs)	1648 (vs)	—20	$\nu(C{=}N)$

But even in this case a sufficiently large steric effect is involved in the formation of a donor—acceptor complex, especially with i-Bu_4Al_2O, where the bulky i-Bu groups eventually exclude the acceptor possibilities.

The strongest of the considered Lewis bases was benzylidene-n-butylamine (SB). It has the highest pK_a value (6.7) and also the greatest influence on the aluminoxane, from PMR, during complexation. The formation of complex IV in this case is confirmed by IR spectral results and cryoscopic molecular weight determinations. In the IR spectrum of an Et_4Al_2O + SB equimolar mixture a strong band at 1628 cm^{-1}, attributable to C=N stretching vibrations of complexed base, appears ($\nu(C{=}N)$ = 1648 cm^{-1} in a free compound) [13]. The measured molecular weight of the complex agrees well with the theoretical calculations of a 1/1 composition and proves additionally its monomeric structure in benzene solution (Tables 3 and 4).

From the sterical point of view benzylidene-n-butylamine, as a Schiff base, occurs in equilibrium between *cis* and *trans* configurations, although the *trans* configuration predominates in solutions [14]. The *cis*—*trans* isomerization of

TABLE 4

THE MOLECULAR WEIGHT MEASUREMENTS OF VARIOUS MIXTURES OF ALUMINOXANES WITH ELECTRON DONORS (Benzene, +5°C)

Components	Molar ratio	Measured mol. wt.	Calculated mol. wt. [a]	Degree of association	Remarks
Et_4Al_2O + PhCN	1/1	286	289	1	completed
	1/2	198	196		complexation
Et_4Al_2O + NMe_2Ph	1/1	274	307	0.9	equilibrium
	1/2	200	214		complex
Et_4Al_2O + SB	1/1	345	345	1	completed
	1/2	254	252		complexation
i-Bu_4Al_2O + PhCN	1/1	351	401	0.88	equilibrium
	1/2	228	252		complex
i-Bu_4Al_2O + NMe_2Ph	1/1	296	419		no complexation

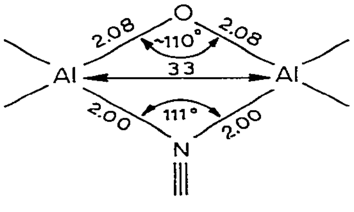
[a] In this column the value corresponding to 1/1 mixture is calculated for monomeric, 1/1 complex, the value corresponding to 1/2 mixture is calculated as the average for the mixture $R_4Al_2O \cdot Do + Do$.

SB ceases as a result of complexation, as was observed earlier [15]. After the formation of a complex with the aluminoxane, the *cis* configuration should be the only one present. The most probable structure of complex IV is the arrangement of two perpendicular planes, one containing Al—O—Al linkage and the other containing the PhHC=N-n-Bu molecule.

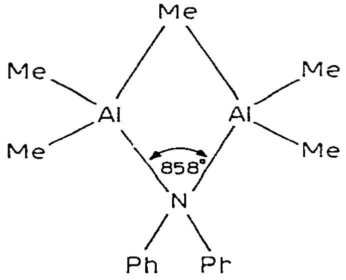
Decreasing the temperature in PMR investigations to —70°C or even lower [7] does not change in principle the character of the spectra of all the investigated complexes. It means, as was suggested in our recent report [7] and also earlier [1], that the donor heteroatom is attached to both aluminium atoms in the complex molecule. The formed bond is probably (for nitrogen) an electron deficient one, which is very well known for carbon in organoaluminium chemistry [16]. A review of papers concerning organoaluminium—nitrogen bonds in four-membered ring systems, non electron deficient [17] or mixed electron deficient-non electron deficient [18,19] allows us to give an approximate description of the geometry of the electron deficient bridging nitrogen bond.

The basic structural feature of the complex between aluminoxane and nitrogen containing donor molecule is the four-membered heterocyclic ring system composed of two aluminium atoms, one oxygen atom and one nitrogen atom. Oxygen forms two σ-bonds with both aluminium atoms. The observed vibrations of the Al—O—Al system in IR agree well with the calculated ones for the value of the Al—O—Al angle 110° and Al—O bond length 2.08 Å [1]. The calculated Al—Al distance in this case equals 3.4 Å and is similar to that in aluminium suboxide Al_2O (3.2 Å) [20]. The Al—N bond length taken from Pauling's sum of covalent radii [21] and compared with the results of X-ray studies of similar systems is on average 2.00 Å. By these assumptions the value of the Al—N—Al bridging bond angle in our case is equal to 111° (V).

The value of the angle Al—N—Al in structure V differs from that determined for the system VI(85.8°) [18,19]. However, in other organoaluminium—nitrogen systems, e.g. $(Me_2AlNHMe)_3$ trimer (VII), the value 122.3° for the Al—N—Al angle was determined [22] with the Al—N bond length practically (1.953 Å) the same as in four-membered rings. The value of 111° calculated by us lies between the two mentioned above, thus the structure V is geometrically probable. But one has to remember that the nitrogen bridge in our case is an electron deficient one and therefore it may differ from the non electron deficient one, in both the Al—N—Al angle and Al—N bond length. Also, the geometry around both aluminium atoms can be distorted from tetrahedral symmetry [19].



(V)



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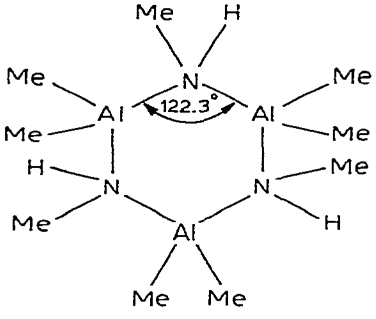
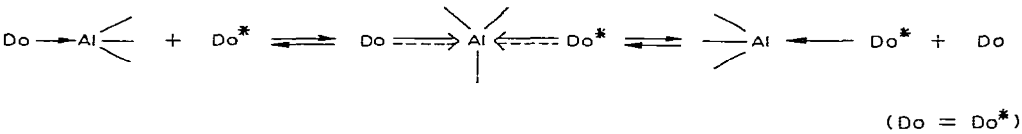


(VII)

The introduction of a donor excess to a solution containing monocoordinate aluminoxane complex does not cause, in any case, the formation of the corresponding dicoordinated one. In the PMR spectra of the aluminoxane: donor mixtures of 1/2 molar ratio, the observed chemical shifts of aluminoxane alkyl grou protons do not change when compared with a mixture of 1/1 molar ratio. It means that the second donor molecule does not take part in complexation. The mixture of uncomplexed donor with the corresponding monocoordinated complex shows one set of characteristic donor proton resonances at a chemical shift which is a weighted mean of that expected for uncomplexed and complexed donor. This is indicative of fast exchange of complexed donor for uncomplexed donor (on the NMR time scale). In the IR spectra of 1/2 aluminoxane/donor mixtures the two bands appear in the characteristic donor region; one of complexed and the other of uncomplexed donor. The measured molecular weight of these mixtures are lower when compared with the 1/1 mixtures; in some cases they are exactly the mean between the molecular weight of monocoordinated complex and the donor molecular weight.

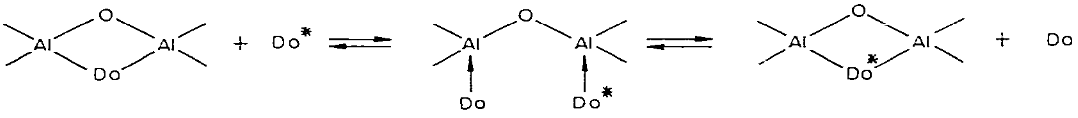
We have observed similar behaviour in PMR and IR spectral studies of triethylaluminium/donor 1/2 mixtures (Table 1). Similar results were found by Mole [23] in the case of triphenylaluminium etherate in the presence of an excess of ether. Some amine complexes gave the same effect, although in this case the PMR spectra showed two resonances attributable to complexed and uncomplexed amine, since base exchange was here much slower. The bimolecular mechanism, sensitive to steric effects, was suggested to be general for the displacement of complexed base [16], probably according to Scheme 1. It is very probable that the donor-exchange mechanism in the case of aluminoxane complexes, in the presence of donor excess, will also be the bimolecular one. The bidentate electron-accepting nature may be employed in the transition state of this exchange, when one Al—O—Al linkage should coordinate actually two donor molecules. Such arrangements, with relatively longer half-lives were observed [5]. Also,

Scheme 1



stable complexes of chloromethylaluminoxanes with benzonitrile with the donor—acceptor molar ratio greater than 1 were obtained [6]. In the associated form of the diethyl ether [2] and benzonitrile [6] complexes with chloromethylaluminoxanes, the two Al atoms of the Al—O—Al system are coordinated by two different donor atoms: the aluminoxane oxygen atom and the donor heteroatom. Based upon this we suggest the following scheme for the description of the exchange mechanism (Scheme 2).

Scheme 2



(Do = Do*)

The results obtained and the literature data lead us finally to some generalizations in the field of aluminoxane complex chemistry:

(*i*) Steric factors decide mainly the possibilities of the complex formation and the complex association. The donor—acceptor properties must be considered a sum of electronic (base strength, aluminoxane acidity) and steric effects.

(*ii*) Arrangements which offer less steric hindrance to complex formation admit the complex association and the coordination of more than one donor molecule per Al—O—Al linkage. The complex association should be considered as a factor donating the second aluminium atom. Acid—base behaviour has, in these cases, a greater influence on the complex formation.

(*iii*) It is possible to synthetize complexes between sterically hindered arrangements by another route: a partial hydrolysis of the corresponding complexed trialkylaluminium.

(*iv*) The basic structural feature of monomeric 1/1 complexes is the four-membered heterocyclic ring composed of two aluminium atoms, one oxygen atom and one donor heteroatom. In the case of nitrogen the electron deficient three centre bond occurs.

(*v*) The donor excess causes its fast exchange according to bimolecular mechanism. This exchange is not hindered at low temperatures.

Experimental

All the experiments were carried out in a purified nitrogen atmosphere. The reactants and solvents were deoxidized and dried immediately before use.

All the studied complexes were prepared in the following way: to the weighed sample of a standard aluminoxane solution (g) the determined amount (by volume, μl) of the liquid donor was added with the aid of Hamilton syringe. The solution was then stirred at room temperature.

IR spectra were recorded in the 2300—600 cm^{-1} range on a Perkin-Elmer Model 577 spectrometer as liquid films between KBr plates. NMR spectra were recorded using a Jeol-JNM-4H-100 apparatus from ca. 10 wt.% methylene chloride, benzene or toluene solutions.

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