

Dimethylaluminum and gallium amino alkoxides

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Abstract

Dimethylaluminum and gallium alkoxides of the type Me_2MOR^* were synthesized ($\text{M} = \text{Al}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (1), $\text{CH}(\text{CH}_3)\text{CH}_2\text{NH}_2$ (2), $\text{M} = \text{Ga}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (3)) by reaction of Me_3M ($\text{M} = \text{Al}$; Ga) with the corresponding amino alcohols. The resulting compounds 1–3 have been characterised by ^1H , ^{13}C and ^{27}Al NMR spectroscopy. The crystal structures of 1–3 were determined by X-ray diffraction. Compounds 1–3 were proved to be dimeric in solid state containing five-membered heterocyclic rings. 3 crystallises in the monoclinic space group $P2_1/n$ with $Z = 4$, $a = 7.898(2)$, $b = 15.512(9)$, $c = 8.453(2)$ Å, and $\beta = 103.70(2)^\circ$, $V = 1000.1(9)$ Å³. The Ga–N distance in 3 runs to 2.525(2) Å which is significantly longer than the Al–N distances in the investigated Al compounds 1 and 2 (2.246(2) and 2.172(3) Å).

Zusammenfassung

Dimethylaluminium- und Dimethylgalliumalkoxide der allgemeinen Formel Me_2MOR^* wurden durch Umsetzung von Me_3M ($\text{M} = \text{Al}$; Ga) mit den entsprechenden Aminoalkoholen synthetisiert ($\text{M} = \text{Al}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (1), $\text{CH}(\text{CH}_3)\text{CH}_2\text{NH}_2$ (2), $\text{M} = \text{Ga}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (3)) und mittels ihrer ^1H -, ^{13}C - und ^{27}Al -NMR-Spektren charakterisiert. Die Kristallstrukturen von 1–3 wurden bestimmt. Die Verbindungen 1–3 liegen im festen Zustand als verbrückte Dimere vor, die 5gliedrige Heterocyclen enthalten. 3 kristallisiert in der monoklinen Raumgruppe $P2_1/n$ mit $Z = 4$, $a = 7.898(2)$, $b = 15.512(9)$, $c = 8.453(2)$ Å und $\beta = 103.70(2)^\circ$, $V = 1000.1(9)$ Å³. Der Ga–N Abstand (2.52 Å) in 3 ist signifikant länger als die Al–N-Bindungslängen in 1 und 2 (2.246(2) und 2.172(3) Å). © 1997 Published by Elsevier Science S.A.

Keywords: Aluminium; Gallium; Amino alkoxide; X-ray diffraction; Chirality

1. Introduction

Simple dimethylaluminum alkoxides Me_2AlOR ($\text{R} = \text{CH}_3$; CH_3CH_2 ; [1], $\text{CH}(\text{CH}_3)_2$; $\text{C}(\text{CH}_3)_3$; C_6H_5 [2]) have been known for many years. The alkoxide groups act as bridging ligands between two dimethylaluminum moieties resulting in dimeric compounds with planar Al_2O_2 rings. Me_2AlOMe is the only exception with a trimeric structure [3,4]. The compounds do not undergo disproportionation in solvents like benzene and diethylether [2].

$[\text{Me}_2\text{AlOCH}_2\text{CH}_2\text{NMe}_2]_2$ was first described by Hoffmann in 1967 as a dimethylaluminum amino alkoxide with the N-atom in β -position [5]. Optically active dialkylaluminum alkoxides (dialkylaluminum-l-

mentholates and -l-borneolates) were first synthesized by Oliver and coworkers with nearly planar Al_2O_2 ring systems in solid state [6].

Dialkylaluminum alkoxides with chiral amino alkoxide groups are of special interest. Typical compounds of this type were obtained by reactions of trialkylaluminum compounds with the corresponding alcohols. Oliver and coworkers reported on the synthesis and characterisation of the monomer dimethylaluminum(2S,3R)-(+)-4-(dimethylamino)-1,2-diphenyl-3-methyl-2-butoxide and the dimer dimethylaluminum(S)-(–)-1-methyl-2-pyrrolidinylmethoxide with Al_2O_2 and AlOC_2N ring systems. These compounds retain their optical activity [7].

Trimethylgallium reacts with alcohols in almost the same manner as trimethylaluminum forming dimethylgallium alkoxides, which structures correspond to those of the dimethylaluminum alkoxides. Dimethylgallium

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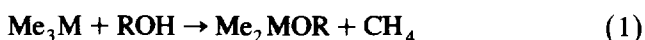
methoxide exhibits, unlike the trimeric aluminum compound, a dimeric structure [8].

Me_2AlOMe and Me_2AlOEt have been proved to be active catalysts, i.e. in the polymerisation of lactones with formation of polyesters [9,10]. $[\text{Me}_2\text{AlOCH}_2\text{CH}_2\text{NMe}_2]_2$ was used to catalyse the ring-opening polymerisation of ϵ -caprolactone to polycaprolactone [11].

Therefore we tried to get more detailed information about the synthesis and structures of other related compounds. We synthesized dimethylaluminum and dimethylgallium compounds with chiral amino alkoxide groups and report on their properties and structures.

2. Results and discussion

Trimethylaluminum and trimethylgallium were reacted with racemic dimethylamino-2-propanol and (S)-(+)-amino-2-propanol in molar ratio 1:1. The corresponding alkoxides Me_2AlOR^* and Me_2GaOR^* were formed according to Eq. (1). The reactions proceed smoothly in *n*-pentane at room temperature.



($\text{M} = \text{Al}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (1), $\text{CH}(\text{CH}_3)\text{CH}_2\text{NH}_2$ (2), $\text{M} = \text{Ga}$; $\text{R} = \text{CH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ (3)).

1–3 are colourless crystalline solids, which can be sublimed in vacuum or recrystallised from toluene. They are very well soluble in aromatic, less in aliphatic hydrocarbons and sensitive to moisture and oxygen. Surprisingly, 2 decomposes above 100°C with evolution of methane. No reaction between the NH_2 groups

and the Me_2Al units can be observed at lower temperatures.

The ^1H , ^{13}C , and ^{27}Al NMR spectra of 1–3 are listed in Table 1. In the ^1H NMR spectra of 1–3 the absence of the $-\text{OH}$ resonance of bifunctional amino alcohols, and the presence of a sharp upfield resonance (1 – 0.58, 2 – 0.60, 3 – 0.07 ppm) for the $-\text{MMe}_2$ moiety of Me_2MOR^* , in the appropriate integration provide good evidence for the O–M coordination in the compounds. The $(\text{CH}_3)_2\text{N}$ signals of 1 and 3 (1.86, 1.84 ppm) are shifted to lower field in comparison with the signal of the corresponding aminoalcohol (1.72 ppm), which indicates a coordinative M–N interaction. A similar situation was found in the case of 2, with a broad signal at 0.78 ppm, which is typical for compounds with coordinated NH_2 groups. In consequence of the presence of an asymmetric carbon atom in all compounds, two multiplets of equal intensity (1H) appear for the neighbouring CH_2 groups. In case of 2, which is the pure (S)-enantiomer, we find two signals for this group of equal intensity.

The ^{13}C NMR spectra of the Me_2MOR^* compounds also show the presence of appropriate resonances which are slightly highfield shifted from the corresponding signals in the parent alcohols and thus support the formation of complexes. 1–3 show high field signals of low intensity for the CH_3 groups attached to the metal atoms.

The ^{27}Al NMR spectra of 1 and 2 indicate, in comparison with the situation in other organoaluminum compounds [12], five-fold coordinated Al atoms. Therefore we consider 1 and 2 as dimeric complexes with a central Al_2O_2 ring and two AlOC_2N chelate rings, according to Figs. 1 and 2 and similar to the structures of other dialkylaluminum alkoxides [13].

Table 1
 ^1H , ^{13}C and ^{27}Al NMR spectra of 1–3 (C_6D_6 , 22°C, [ppm])

Compound	^1H		^{13}C		^{27}Al
$\text{Me}_2\text{AlOCH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ 1	–0.58 (s)	$(\text{CH}_3)_2\text{Al}-$	–9.25	$(\text{CH}_3)_2\text{Al}-$	124.5
	1.12 (d)	$\text{CH}_3\text{C}-$	21.09	$\text{CH}_3\text{C}-$	
	1.47 (m)	$-\text{NCH}_2-$	45.25	$(\text{CH}_3)_2\text{N}-$	
	2.19 (t)		64.90	$-\text{NCH}_2-$	
	1.86 (s)	$(\text{CH}_3)_2\text{N}-$	66.99	$-\text{OCH}-$	
	3.76 (m)	$-\text{OCH}-$			
$\text{Me}_2\text{AlOCH}(\text{CH}_3)\text{CH}_2\text{NH}_2$ 2	–0.60 (s)	$(\text{CH}_3)_2\text{Al}-$	–6.61	$(\text{CH}_3)_2\text{Al}-$	125.3
	0.78 (m,br)	$-\text{NH}_2$	20.99	$\text{CH}_3\text{C}-$	
	1.30 (d)	$\text{CH}_3\text{C}-$	46.85	$-\text{NCH}_2-$	
	1.71 (m)	$-\text{NCH}_2-$	64.88	$-\text{OCH}-$	
	1.93 (m)				
	3.57 (m)	$-\text{OCH}-$			
$\text{Me}_2\text{GaOCH}(\text{CH}_3)\text{CH}_2\text{NMe}_2$ 3	–0.07 (s)	$(\text{CH}_3)_2\text{Ga}-$	–4.45	$(\text{CH}_3)_2\text{Ga}-$	
	1.10 (d)	$\text{CH}_3\text{C}-$	22.58	$\text{CH}_3\text{C}-$	
	1.59 (q)	$-\text{NCH}_2-$	45.56	$(\text{CH}_3)_2\text{N}-$	
	2.04 (t)		63.76	$-\text{NCH}_2-$	
	1.84 (s)	$(\text{CH}_3)_2\text{N}-$	68.19	$-\text{OCH}-$	
	3.81 (m)	$-\text{OCH}-$			

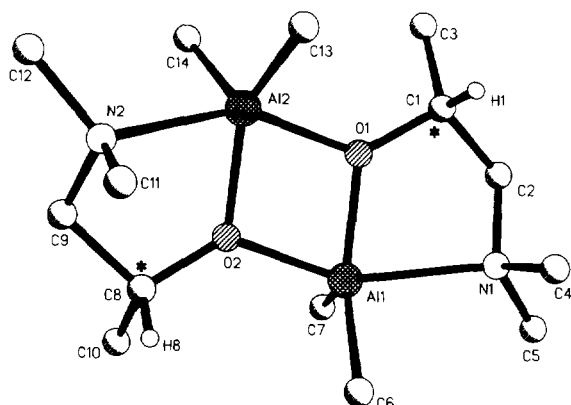


Fig. 1. Molecular structure of **1** (hydrogen atoms with exception of H1 and H8 have been omitted for clarity).

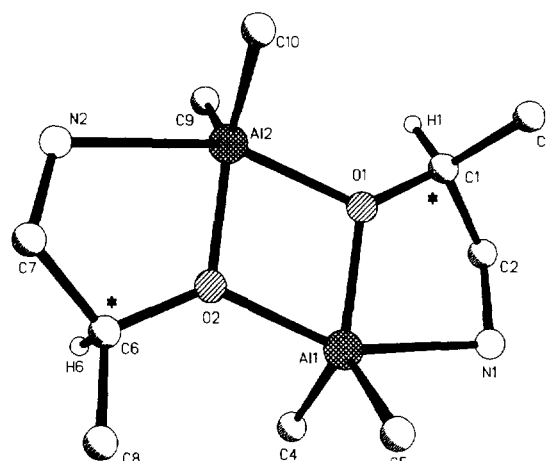


Fig. 2. Molecular structure of **2** (hydrogen atoms with exception of H1 and H6 have been omitted for clarity).

Single crystals of **1** and **3** were obtained by sublimation in vacuum (66°C/3 Torr, 69°C/3 Torr) and of **2** by cooling a saturated toluene solution. The crystal data and details of X-ray structure determination are listed in Table 2, the atomic coordinates, equivalent isotropic displacement parameters in Table 3 and selected bond lengths and angles in Table 4. In solid state the compounds **1–3** form dimeric molecules (Figs. 1–3). The central four-membered Al_2O_2 and Ga_2O_2 rings are nearly planar and each metal atom is coordinated in a distorted trigonal bipyramidal fashion by two methyl groups in axial, two alkoxide substituents in axial and equatorial and the N atom of the aminoalkoxide group in equatorial position.

Haaland introduced the division into normal, dative and intermediate bonds to describe donor–acceptor compounds [14]. In the investigated aluminum and gallium compounds two bonds of intermediate type are formed between the metal and the O atoms. Thus, the equatorial Al1–O1 and Ga1–O1A 3). The Al–N bonds can be described as purely dative. The Ga1–N1 bond in **3** is much weaker than the Al1–N1 bond in the analogous compound **1**. The M–O and M–N distances are similar to those found in analogous structures of five-coordinated aluminum and gallium atoms [6,7,15].

Table 2
Crystallographic data and experimental parameters of **1–3**

Compound	1	2	3
Formula	$\text{C}_{14}\text{H}_{36}\text{O}_2\text{N}_2\text{Al}_2$	$\text{C}_{10}\text{H}_{28}\text{O}_2\text{N}_2\text{Al}_2$	$\text{C}_{14}\text{H}_{36}\text{O}_2\text{N}_2\text{Ga}_2$
Formula weight [g mol^{-1}]	318.4	262.3	403.84
Crystal	platelet, colourless	prism, colourless	platelet, colourless
Crystal size [mm^3]	$0.44 \times 0.3 \times 0.25$	$0.5 \times 0.25 \times 0.25$	$0.25 \times 0.4 \times 0.6$
Crystal system	triclinic	orthorhombic	monoclinic
Space group; Z	$P\bar{1}$; 2	$P2_12_12_1$; 2	$P2_1/n$; 4
a [\AA]	7.216(2)	8.782(1)	7.898(2)
b [\AA]	9.576(3)	11.853(2)	15.512(9)
c [\AA]	15.862(7)	15.108(2)	8.453(2)
α [$^\circ$]	80.35(2)	90	90
β [$^\circ$]	88.94(2)	90	103.70(2)
γ [$^\circ$]	71.32(2)	90	90
Volume [\AA^3]	1023.1(6)	1572.7(4)	1006.1(9)
Density (calc.) [mg m^{-3}]	1.034	1.108	1.333
Absorption coeff. [mm^{-1}]	0.146	0.177	2.681
θ range [$^\circ$]	2.28 to 27.04	2.18 to 26.03	2.63 to 30.04
Reflections/ R_{int}	4853/0.066	3588/0.018	4128/0.014
Data/parameters	4492/325	3115/214	2936/133
Goof on F^2	1.016	1.088	1.021
R_1/wR_2 [$I > 2\sigma(I)$]	0.034/0.091	0.038/0.096	0.044/0.120
R_1/wR_2 [all data]	0.066/0.104	0.058/0.102	0.058/0.129
Largest diff. peak [e \AA^{-3}]	0.275	0.497	1.158 (near Ga1)

The asymmetric carbon atoms of OR* are C1 and C8 in compound **1**, C1 and C6 in **2**, C1 in **3**. In the structure of **3** ($P2_1/n$) the centre of the Ga_2O_2 ring coincides with a crystallographic inversion centre, and each molecule contains one (R) and one (S) enantiomeric form of Me_2GaOR^* .

Table 3

Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$] of 1–3

Atom	x	y	z	U_{eq}
[Me₂AlOCH(CH₃)CH₂NMe₂]₂ 1				
Al(1)	4889(1)	7147(1)	7474(1)	31(1)
Al(2)	7615(1)	4086(1)	7340(1)	31(1)
O(1)	6118(2)	5990(1)	6673(1)	32(1)
O(2)	6607(2)	5331(1)	8133(1)	32(1)
N(1)	3080(2)	8398(2)	6282(1)	41(1)
N(2)	8045(3)	2450(2)	8566(1)	54(1)
C(1)	5687(3)	6422(2)	5776(1)	35(1)
C(2)	4536(3)	8081(2)	5622(1)	41(1)
C(3)	7528(3)	6093(3)	5267(2)	51(1)
C(4)	1514(3)	7770(3)	6141(2)	57(1)
C(5)	2223(4)	10030(2)	6246(2)	65(1)
C(6)	2550(3)	7119(3)	8145(1)	49(1)
C(7)	6114(4)	8680(2)	7598(2)	56(1)
C(8)	6883(3)	4865(2)	9033(1)	38(1)
C(9)	8434(3)	3329(2)	9179(1)	52(1)
C(10)	7485(3)	5959(2)	9460(1)	49(1)
C(11)	6224(5)	2087(3)	8766(2)	80(1)
C(12)	9685(6)	1050(3)	8585(2)	90(1)
C(13)	6503(3)	2863(2)	6734(1)	47(1)
C(14)	10388(3)	3902(3)	7155(2)	50(1)
[Me₂AlOCH(CH₃)CH₂NH₂]₂ 2				
Al(1)	1162(1)	2804(1)	2202(1)	28(1)
Al(2)	3888(1)	1631(1)	2956(1)	33(1)
O(2)	2963(2)	1954(2)	1885(1)	35(1)
O(1)	2175(2)	2579(1)	3266(1)	32(1)
N(1)	-442(3)	3529(2)	3136(2)	38(1)
N(2)	5346(3)	673(3)	2061(2)	51(1)
C(5)	1479(3)	4227(2)	1528(2)	42(1)
C(4)	-391(3)	1704(2)	1792(2)	42(1)
C(2)	-192(3)	2906(3)	3963(2)	40(1)
C(1)	1527(3)	2829(2)	4104(2)	34(1)
C(3)	2180(4)	3918(3)	4482(2)	44(1)
C(9)	3362(4)	274(2)	3658(2)	50(1)
C(10)	5637(3)	2602(2)	3302(2)	44(1)
C(7)	5099(4)	1147(3)	1190(2)	60(1)
C(6)	3424(3)	1408(3)	1090(2)	44(1)
C(8)	3087(5)	2067(4)	282(2)	65(1)
[Me₂GaOCH(CH₃)CH₂NMe₂]₂ 3				
Ga(1)	297(1)	9622(1)	6782(1)	37(1)
O(1)	1165(3)	9490(2)	4860(2)	48(1)
N(1)	2699(3)	8519(2)	7585(3)	44(1)
C(1)	2796(5)	9108(3)	4941(4)	61(1)
C(2)	3077(5)	8350(3)	6027(4)	69(1)
C(3)	3079(7)	8916(4)	3253(5)	72(1)
C(4)	4248(5)	8861(3)	8738(5)	69(1)
C(5)	2181(7)	7730(3)	8295(8)	86(1)
C(6)	1512(6)	10443(2)	8434(5)	57(1)
C(7)	-1564(5)	8810(3)	6942(6)	60(1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

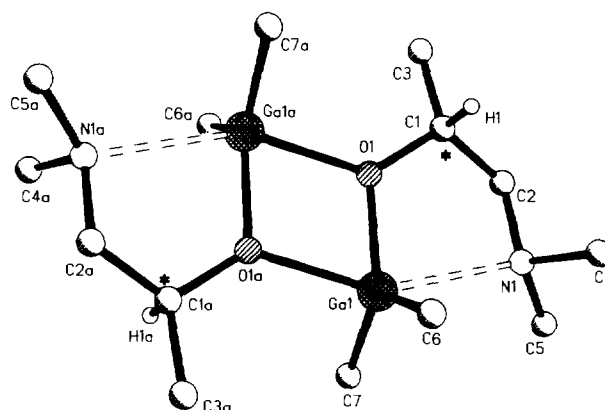


Fig. 3. Molecular structure of **3** (hydrogen atoms with exception of H1 and H1a have been omitted for clarity).

In the structure of **1** the asymmetric unit comprises a complete dimer. The space group was found to be $P\bar{1}$, but one dimer consists of two Me_2AlOR^* of the identical enantiomeric form (R,R or S,S). A second kind of molecule within the crystal structures is generated by the centres of symmetry. That means that the crystal lattice is built up of two different kinds of molecules consisting of either the (S) or the (R) enantiomeric form of Me_2AlOR^* exclusively.

In the crystal structure of **2** ($P2_12_12_1$; Flack parameter 0.04), the situation is quite different. One dimeric complex, always containing solely the (S) enantiomeric form of Me_2AlOR^* , is identical with the asymmetric unit. Apart from van der Waals forces, no intermolecular interactions could be found in the presented crystal structures.

3. Experimental

3.1. General procedure

Me_3Al , Me_3Ga and the amino alcohols were used in the form of commercial products. All operations were performed under an argon atmosphere using normal Schlenk techniques. Solvents were dried by treatment with LiAlH_4 . After distillation they were saturated with argon. The amino alcohols were freshly distilled before use.

3.2. X-ray crystal structure determination of 1–3

Intensity data were measured by means of a Stoe STADI4 diffractometer using graphite monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$; ω - θ mode) at a temperature of 200 K. An empirical absorption correction was applied for **3**. The structures were determined by direct methods using SHELXS [16] and full-matrix least-squares refinement based on F^2 (SHELXS-93 [17]).

Table 4
Selected bond lengths [Å] and angles [°] of the compounds 1–3

1		2		3 ^a	
<i>Bond lengths [Å]</i>					
Al1–O1	1.852(1)	Al1–O1	1.856(2)	Ga1–O1	1.919(2)
Al2–O2	1.856(1)	Al2–O2	1.851(2)		
Al1–O2	1.927(1)	Al1–O2	1.936(2)	Ga1–O1a	2.097(2)
Al2–O1	1.936(1)	Al2–O1	1.936(2)		
Al1–N1	2.246(2)	Al1–N1	2.172(3)	Ga1–N1	2.525(2)
Al2–N2	2.241(2)	Al2–N2	2.181(3)		
Al1–C6	1.984(2)	Al1–C4	1.986(3)	Ga1–C6	1.964(3)
Al1–C7	1.977(2)	Al1–C5	1.990(3)	Ga1–C7	1.964(4)
Al2–C13	1.986(2)	Al2–C9	1.981(3)		
Al2–C14	1.973(2)	Al2–C10	1.988(3)		
<i>Bond angles [°]</i>					
O1–Al1–C6	130.1(1)	O1–Al1–C4	120.3(1)	O1–Ga1–C6	117.3(2)
O1–Al1–C7	111.8(1)	O1–Al1–C5	119.9(1)	O1–Ga1–C7	114.4(2)
O2–Al2–C13	132.7(1)	O2–Al2–C9	122.2(1)		
O2–Al2–C14	110.4(1)	O2–Al2–C10	116.7(1)		
O1–Al1–N1	78.7(1)	O1–Al1–N1	78.8(1)	O1–Ga1–N1	76.1(1)
O2–Al2–N2	77.9(1)	O2–Al2–N2	79.8(1)		
O2–Al1–N1	151.0(1)	O2–Al1–N1	153.8(1)	O1a–Ga1–N1	152.1(1)
O1–Al2–N2	148.6(1)	O1–Al2–N2	155.3(1)		
O2–Al1–C6	94.9(1)	O2–Al1–C4	98.1(1)	O1a–Ga1–C6	98.3(1)
O2–Al1–C7	104.2(1)	O2–Al1–C5	101.6(1)	O1a–Ga1–C7	99.4(2)
O1–Al2–C13	95.5(1)	O1–Al2–C9	99.2(1)		
O1–Al2–C14	105.8(1)	O1–Al2–C10	101.6(1)		
N1–Al1–C6	92.9(1)	N1–Al1–C4	91.0(1)	N1–Ga1–C6	98.3(2)
N1–Al1–C7	96.5(1)	N1–Al1–C5	95.0(1)	N1–Ga1–C7	99.4(2)
N2–Al2–C13	92.1(1)	N2–Al2–C9	92.6(1)		
N2–Al2–C14	97.8(1)	N2–Al2–C10	90.6(1)		
O1–Al1–O2	74.9(1)	O1–Al1–O2	75.4(1)	O1–Ga1–O1a	76.2(1)
O2–Al2–O1	74.6(1)	O2–Al2–O1	75.5(1)		
C6–Al1–C7	118.1(1)	C4–Al1–C5	119.5(1)	C6–Ga1–C7	127.9(2)
C13–Al2–C14	116.8(1)	C9–Al2–C10	120.6(1)		

^a Symmetry transformation used to generate equivalent atoms in the structure of 3: $-x, 2-y, 1-z$.

Details are listed in Table 2. Further details of the structure investigation are available on request from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen 2, Germany, CSD No. 486646 (1), 476646 (2) and 406646 (3).

3.3. Dimethylaluminum-(+);(-)-dimethylamino-2-propoxide (1)

To a solution of 3.6 g (4.8 ml; 0.05 mol) trimethylaluminum in 50 ml n-pentane, 5.1 g (6.2 ml; 0.05 mol) (+);(-)-dimethylamino-2-propanol were added dropwise at a temperature of -15°C . After warming to room temperature, the solvent was removed in vacuum. The residue was sublimed in vacuum ($66^{\circ}\text{C}/3\text{Torr}$) creating transparent colourless crystals (yield 6.4 g (80%)).

Anal. Found: C, 52.70; H, 10.38; N, 8.45. $\text{C}_7\text{H}_{18}\text{ONAl}$ Calc.: C, 52.86; H, 10.32; N, 8.80%. NMR data see Table 1.

3.4. Dimethylaluminum-(S)-(+)-amino-2-propoxide (2)

According to the synthesis of 1, 1.8 g (2.2 ml; 0.03 mol) Me_3Al was treated with a solution of 2.2 g (0.03 mol) (S)-(+)-amino-2-propanol in 50 ml n-pentane. The white precipitate obtained was recrystallised from toluene. The crystals were filtered at -78°C and dried in vacuum (yield 2.7 g (77%)). Anal. Found: C, 45.70; H, 10.17; N, 10.54. $\text{C}_5\text{H}_{14}\text{ONAl}$ Calc.: C, 45.80; H, 10.69; N, 10.67%. NMR data see Table 1.

3.5. Dimethylgallium-(+);(-)-dimethylamino-2-propoxide (3)

According to the synthesis of 1, 2.6 g (3.0 ml; 0.025 mol) (+);(-)-dimethylamino-2-propanol in 20 ml n-pentane were added dropwise to 1.9 g (2.5 ml; 0.025 mol) Me_3Ga in 50 ml of n-pentane at -20°C . After warming up to room temperature the solvent was removed and the residue sublimed in vacuum

(69 °C/3 Torr) forming transparent colourless crystals (yield 3.9 g (78%)). Anal. Found: C, 46.25; H, 9.12; N, 6.12. $C_7H_{18}ONGa$ Calc.: C, 46.30; H, 9.35; N, 6.89%. NMR data see Table 1.

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