

HETEROCYCLIC COMPOUNDS OF ALUMINIUM (III) WITH GLYCOLS:

PART 1 - REACTION OF $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ WITH MOCH_3 (M= Li, Na, K) IN 1:1 MOLAR RATIO

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Abstract

Reaction of $\text{Al}(\text{OPr}^i)_3$ with $\text{HOC}(\text{CH}_3)_3\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ in 1:1 and 1:2 molar ratios in refluxing benzene, have resulted in the synthesis of $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$ and $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$, respectively. These are soluble in a variety of organic solvents (e.g. benzene, chloroform and dimethylsulfoxide). The 1:1 product $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$ exhibits dimeric nature in chloroform. While 1:2 product $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ is monomeric in chloroform. Reaction of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ with MOCH_3 (M = Li, Na and K) in 1:1 molar ratio in refluxing methanol yields $[(\text{CH}_3\text{OH})\text{M}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$. These are soluble in methanol and dimethyl sulfoxide. These bimetallic heterocyclic derivatives are monomeric in methanol and have slight ionic character. Plausible structures has been proposed on the basis of elemental analyses, molecular weight measurements, IR, NMR (^1H , ^{13}C and ^{27}Al) spectral studies. ^{27}Al NMR spectra show the presence of four coordinated aluminum site.

Introduction

Synthesis and optical activity of bimetallic heterocyclic derivative, $\text{K}_3[\text{Al}(\text{O}_2\text{C}_6\text{H}_4)_3]$ have been described¹ as long back as 1932. A survey of the literature suggests that the field had been diversified in 1962 by Mehrotra and Mehrotra², who described the synthesis of heterocyclic derivatives of aluminium (III) by the reaction of

aluminium isopropoxide with glycols in various molar ratios. The glycolates of aluminium (III) have received further attention when IR and thermogravimetric studies have been carried out³.

A new orientation was given to this group of compounds, when more sophisticated and latest techniques like ²⁷Al NMR and X-ray diffraction methods have been employed for characterization and highlighting the structure and bonding features of these derivatives. Crystal structures^{4,5} of Me₂Al[O(CH₂)₂OMe]₂, and [Al₂(O₂C₂H₄)₄]⁻² have been elucidated.

In addition to this, more recently Gainsford et.al.⁶ have carried out X-ray diffraction studies of [Al(OCH₂CH₂O)₂(OCH₂CH₂OH)]⁻² and of trimeric [(Al₃(OCH₂CH₂O)₅(OCH₂CH₂OH)]⁻³ encapsulated Na cation. Crystal structures of these derivatives exhibit many interesting features.

Keeping this in view an effort has been made during the course of the present investigation to synthesize and characterize some interesting bimetallic heterocyclic derivatives of aluminium(III) with 2-methy-2,4-pentanediol.

Experimental

All the solvents used during these investigation were of reagent grade and were made anhydrous by standard methods⁷. Aluminium isopropoxide was prepared as described by Mehrotra⁸.

IR spectra were recorded as Nujol mulls on a Perkin-Elmer 577 spectrophotometer in the range 4000-200 cm⁻¹. ¹H NMR spectra were scanned on a Jeol FX 90Q spectrometer in CDCl₃ using TMS as an internal reference. ¹³C NMR spectral studies have been carried out in benzene using D₂O locks, while ²⁷Al NMR spectra were recorded in benzene using Al(NO₃)₃ as standard reference at 23.79 MHz. Molecular weight measurements were carried out on a Knauer Vapour Pressure Osmonmeter in chloroform at 45°C.

1. Reaction of $\text{Al}(\text{OPr}^i)_3$, with $\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ in 1:1 molar ratio in benzene

A mixture of $\text{Al}(\text{OPr}^i)_3$ (2.51g, 12.29mmol) and $\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ (1.48g, 12.59mmol) was refluxed on a fractionating column for about 3% hrs., with simultaneous removal of isopropanol benzene azeotrope. A clear solution was obtained. The progress of the reaction was checked by estimating isopropanol in azeotrope. The excess of solvent was removed under reduced pressure leaving a white shining powder of the type $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$. The product was also purified by re-crystallization from a mixture of n-hexane and dichloromethane. Yield found : 98%, Anal. found : Al, 13.31% Calculated for $\text{C}_{18}\text{H}_{38}\text{O}_6\text{Al}_2$: Al, 13.34%

Similar procedure was adopted for the preparation of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ by the reaction of $\text{Al}(\text{OPr}^i)_3$ (2.22g, 10.87mmol) with $\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ (2.58g, 21.83mmol) in 1:2 molar ratio in refluxing benzene for 5 hrs. The experimental details for this compound are summarized in Table 1.

2. Reaction of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ with LiOCH_3 in 1:1 molar ratio in methanol

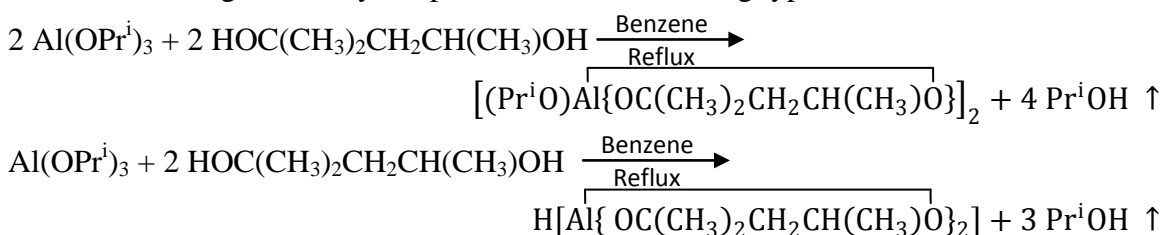
A solution of lithium (0.067g, 9.65mmol) in methanol (~35 ml) was added to a suspension of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ (2.51g, 9.64 mmol) in methanol (~30ml) and the mixture was refluxed for 2 hrs., till a clear solution was obtained. On removing the solvent under reduced pressure a white solid product, $[(\text{CH}_3\text{OH})\text{Li}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ was obtained. The resultant product was further purified by re-crystallization from methanol and benzene mixture. Yield found : 99%. Anal. found : Al, 8.99; C, 51.70; H, 9.37%. Calculated for $\text{C}_{13}\text{H}_{28}\text{O}_5\text{AlLi}$: Al, 9.04; C, 52.34; H, 9.46%.

The other alkali metal derivatives were prepared using a similar procedure and details are summarized in Table - 1.

Results and Discussion

Compounds of 2-methyl-2,4-pentanediol

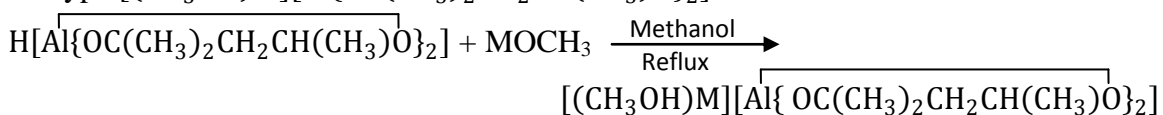
The reaction of $\text{Al}(\text{OPr}^i)_3$, with $\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ in 1:1 and 1:2 molar ratios in refluxing benzene yield products of the following types :



These replacement reactions are straight forward up to the liberation of two moles of the isopropanol after that the later two reactions become comparatively slow and are pushed to completion by continuously removing the liberated Isopropanol azeotropically.

All these derivatives are highly soluble in benzene, chloroform and dimethyl sulfoxide. $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$ and $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ are white solids. The 1:1 product, $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$ exhibits dimeric nature in chloroform, while $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ exhibits monomeric nature in chloroform.

Reactions of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ with MOCH_3 ($\text{M} = \text{Li}, \text{Na}$ and K) in 1:1 molar ratio in refluxing methanol have been found to be facile, yielding products of the type $[(\text{CH}_3\text{OH})\text{M}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$



($\text{M} = \text{Li}, \text{Na}$ and K)

These bimetallic heterocyclic derivatives can be obtained after removal of methanol under reduced pressure. They are white to pale yellow solids, soluble in methanol and dimethyl sulfoxide. These bimetallic heterocyclic derivatives are monomeric in methanol and have slight ionic character⁹ in 0.001 M methanol solution.

(Table-2) These derivatives can be re-crystallized unchanged from a mixture of methanol and benzene.

IR Spectra

The IR spectral data for these bimetallic heterocyclic derivatives and the free ligand have been summarized in Table-3. The spectral data for $[(Pr^iO)Al\{OC(CH_3)_2CH_2CH(CH_3)O\}]_2$, show that broad band present in the spectrum of the free ligand at 3366 cm^{-1} due to ν O-H vibration disappears in the above derivative, indicating the formation of aluminium-oxygen bond. The appearance of a broad band at 3356 cm^{-1} in $H[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$ can be ascribed to the -OH group coordinated to aluminium. Presence of a broad band in the IR spectra of other derivative in the region $3350\text{-}3356\text{ cm}^{-1}$ may be assigned to the -OH group of methanol molecule.

The medium intensity band in the region $1044\text{-}1066$ may be assigned to ν C-O vibration¹⁰⁻¹². A medium to weak intensity band in the region $655\text{-}662\text{ cm}^{-1}$ may tentatively be assigned to Al-O stretching vibration.¹³

Table 1 : Reaction of $\text{Al}(\text{OPr}^i)_3$, with $\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ in 1:1 and 1:2 molar ratios and reaction of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ with MOCH_3 (M=Li, Na and K)

S. No.	Reactants (g)		Molar Ratio	Product	Pr ⁱ OH (g) found (calcd.)	Yield %	Analysis % found (calcd.)		
	a	b					Al	C	H
1.	$\text{Al}(\text{OPr}^i)_3$ 2.51	$\text{HOC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{OH}$ 1.48	1:1	$[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	1.46 (1.48)	98	13.31 (13.34)	-	-
2.	2.22	2.58	1:2	$\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	1.85 (1.96)	98	10.46 (10.36)	54.85 (55.36)	9.40 (9.68)
3.	$\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$ 2.51	LiOCH_3 0.067	1:1	$[(\text{CH}_3\text{OH})\text{Li}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	-	99	8.99 (9.04)	51.70 (52.34)	9.37 (9.46)
4.	2.91	NaOCH_3 0.26	1:1	$[(\text{CH}_3\text{OH})\text{Na}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	-	97	8.52 (8.58)	48.92 (49.67)	8.52 (8.98)
5.	2.68	KOCH_3 0.40	1:1	$[(\text{CH}_3\text{OH})\text{K}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	-	98	8.13 (8.16)	46.52 (47.25)	8.72 (8.54)

Table 2 : Properties of $[(Pr^iO)Al\{OC(CH_3)_2CH_2CH(CH_3)O\}]_2$, $H[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$ and $[(CH_3OH)M][Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$ (M = Li, Na and K).

S.No.	Compound	Nature of the product	Molar Conductance $ohm^{-1}cm^2mol^{-1}$ (methanol)	Molecular weight found (calcd.)
1.	$[(Pr^iO)Al\{OC(CH_3)_2CH_2CH(CH_3)O\}]_2$	White shining solid	-	399 (202)
2.	$H[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$	White shining solid	-	246 (260)
3.	$[(CH_3OH)Li][Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$	White solid powder	57	286 (298)
4.	$[(CH_3OH)Na][Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$	White solid powder	73	299 (314)
5.	$[(CH_3OH)K][Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$	Pale yellow solid	78	310 (331)

Table 3 : IR spectral data (cm⁻¹) of [(PrⁱO)Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂, H[Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ and [(CH₃OH)M][Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ (M = Li, Na and K)

S.No.	Compound	v O-H	Glycolic v C-O	Ring vib.	v Al-O
1.	Ligand HOC(CH ₃) ₂ CH ₂ CH(CH ₃)OH	3366 br	1157m	-	-
2.	[(Pr ⁱ O)Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	-	1066m	948m	656m
3.	H[Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	3356 br	1044m	945m	661w
4.	[(CH ₃ OH)Li][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	3354 br	1046m	946w	662w
5.	[(CH ₃ OH)Na][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	3354 br	1046m	955m	656w
6.	[(CH ₃ OH)K][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	3350 br	1052m	957m	655w

br = broad, m = medium, w = weak

Table 4 : ¹H NMR spectral data (δppm) of [(PrⁱO)Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂, H[Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ and [(CH₃OH)M][Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ (M = Li, Na and K)

S.No.	Compound	Glycolate moiety				Isopropoxy group		Methanol	
		-CH ₃	-CH ₂ -	-CH<	-OH	-CH ₃	-OCH<	-CH ₃	-OH
1.	Ligand HOC(CH ₃) ₂ CH ₂ CH(CH ₃)OH	1.30, m (9H)	1.54, d(2H)	4.51, m(1H)	4.29, br(2H)	-	-	-	-
2.	[(Pr ⁱ O)Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	1.23, m (9H)	1.50, d(2H)	4.29, m(1H)	-	1.39, d (6H)	4.52, m (1H)	-	-
3.	H[Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	1.25, m(18H)	1.52, d(4H)	4.25, m(2H)	3.69, br(1H)	-	-	-	-
4.	[(CH ₃ OH)Li][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	1.25, m(18H)	1.50, d(4H)	4.20, m(2H)	-	-	-	3.35, u	3.49, u
5.	[(CH ₃ OH)Na][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	1.27, m(18H)	1.48, d(4H)	4.22, m(2H)	-	-	-	3.35, u	3.49, u
6.	[(CH ₃ OH)K][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	1.27, m(18H)	1.56, d(4H)	4.22, m(2H)	-	-	-	3.35, u	3.49, u

d = doublet, m = multiplet, br = broad, u = unresolved

¹H NMR Spectra

¹H NMR spectra of these complexes and 2-methyl-2,4-pentanediol were taken in CDCl₃ at ambient temperature and data are summarized in Table-4. The signal due to -OH proton appears at δ 4.29 ppm in free 2-methyl-2,4-pentanediol. This signal is found to be absent in all derivatives, except $H[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$ indicating the formation of Al-O bonds. Derivatives of the type $[(CH_3OH)M][Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$ show a signal at δ 3.49 due to -OH proton of the methanol molecule.

Methyl, methylene and methine protons of 2-methyl-2,4-pentanediol moiety appear at δ 1.23 - 1.25, δ 1.48 - 1.56 and δ 4.20 - 4.29 ppm, respectively.

A doublet at δ 1.39 ppm and a multiplet at δ 4.52 ppm in the spectra of $[(Pr^iO)Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$, may be assigned to methyl and methine protons of isopropoxy group, respectively.

¹³C NMR Spectra

The ¹³C NMR spectra of newly synthesized derivatives along with 2-methyl-2,4-pentanediol are summarized in Table-5. Assignments of the peaks have been made by comparison with the parent glycol. There is no notable shifts in various ¹³C nuclei.

In the ¹³C NMR spectra of all these derivatives, the methyl, methylene, methine and carbonyl carbons, are observed at δ 23.68 - 34.67, δ 49.13 - 50.12, δ 65.17 - 66.52 and δ 69.13 - 72.52 ppm, respectively.

In case of $[(Pr^iO)Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_2]$, signals at δ 26.76 and δ 63.71 have been assigned to the methyl and the methine carbons of the isopropoxy group.

²⁷Al NMR Spectra

²⁷Al NMR spectra¹⁴⁻¹⁷ of some of these representative derivatives at 23.79 MHz in benzene are summarized in Table-6. A perusal of Table-6 indicates that ²⁷Al NMR chemical shift values are observed in the range δ +36.58 to +78.82 as a broad hump. This indicates the presence of tetra-coordinated aluminium(III) atom in all these derivatives. (Fig. 1-3)

Table 5 : ¹³C NMR spectral data (δppm) of [(PrⁱO)Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂, H[Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ and [(CH₃OH)M][Al{OC(CH₃)₂CH₂CH(CH₃)O}]₂ (M = Li, Na and K)

S.No.	Compound	Glycolate moiety				Isopropoxy group	
		-CH ₃	>CH ₂ /CH* ₃ OH	-CH<	>C<	-CH ₃	-OCH<
1.	Ligand HOC(CH ₃) ₂ CH ₂ CH(CH ₃)OH	25.41,28.93,32.07	50.44	65.96	71.89	-	-
2.	[(Pr ⁱ O)Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	25.51,28.28,34.67	49.13	66.52	72.52	26.57	63.71
3.	H[Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	24.76,28.01,32.01	49.84	65.61	71.24	-	-
4.	[(CH ₃ OH)Li][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	23.68,27.15,30.51	49.14	65.77	69.13	-	-
5.	[(CH ₃ OH)Na][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	25.03,27.79,32.40	50.11	65.17	70.97	-	-
6.	[(CH ₃ OH)K][Al{OC(CH ₃) ₂ CH ₂ CH(CH ₃)O}] ₂	25.32,27.86,33.02	50.12	65.56	71.12	-	-

Table No. 6 : ^{27}Al NMR Spectral data (δppm) of some representative aluminium (III) derivatives with 2-methyl-2,4-pentanediol

S.No.	Compound	Shift (δ)	Assignment
1.	$[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$	+36.58	Tetrahedral
2.	$\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	+55.42	Tetrahedral
3.	$[(\text{CH}_3\text{OH})\text{Na}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$	+78.82	Tetrahedral

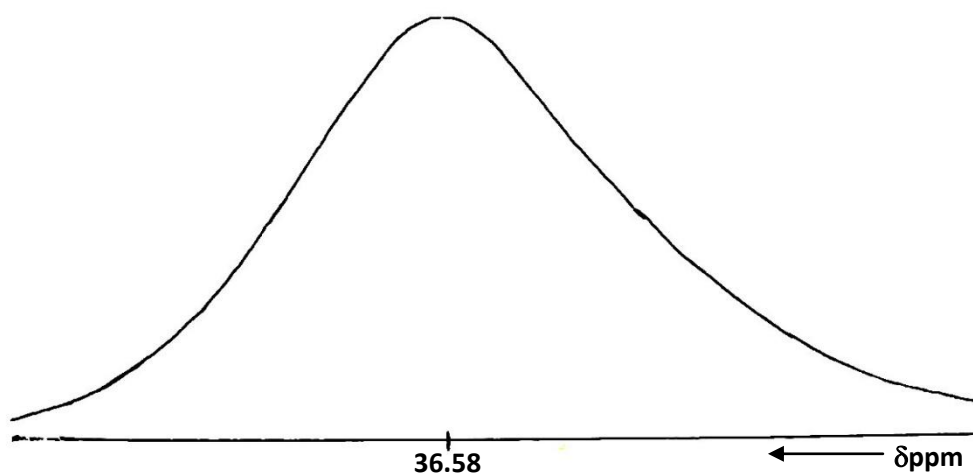


Fig. 1 : ^{27}Al NMR spectrum of $[(\text{Pr}^i\text{O})\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}]_2$

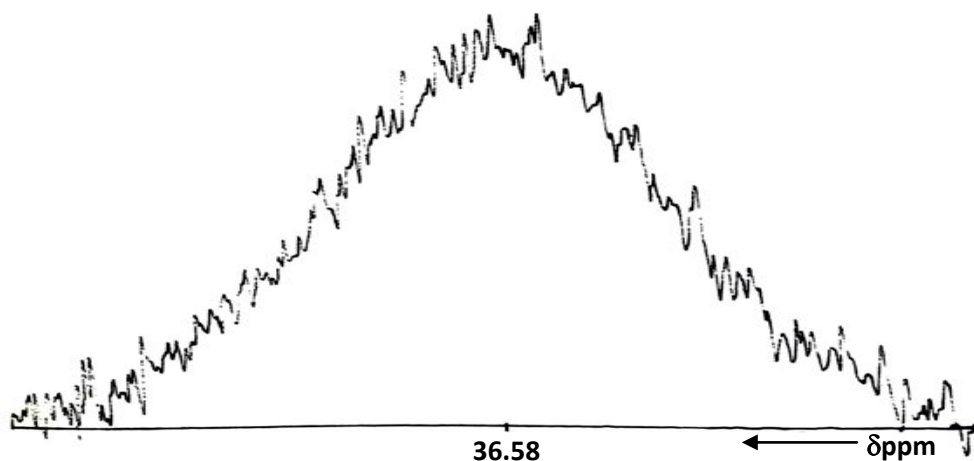


Fig. 2 : ^{27}Al NMR spectrum of $\text{H}[\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$

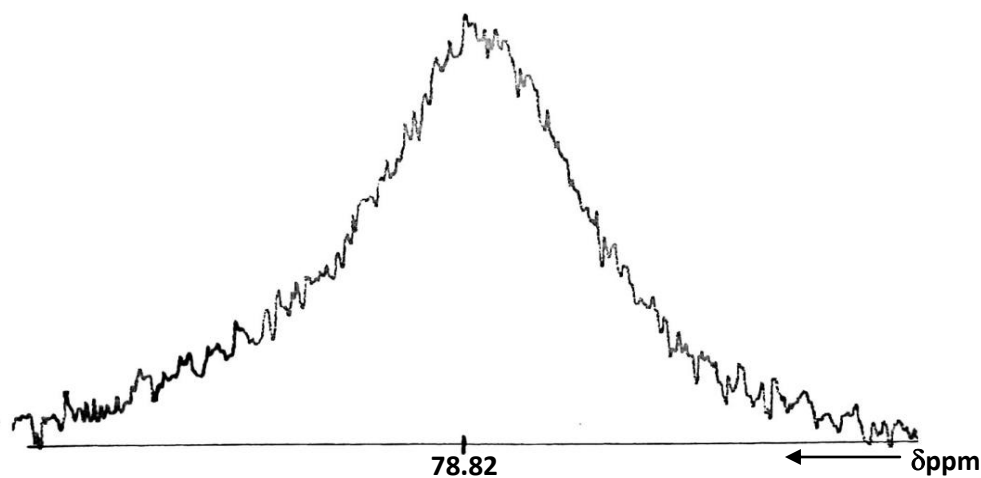


Fig. 3 : ^{27}Al NMR spectrum of $[(\text{CH}_3\text{OH})\text{Na}][\text{Al}\{\text{OC}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{O}\}_2]$

55.42 δppm

Structural Features

In the absence of single crystal X-ray analysis of at least one of the representative heterocyclic glycolates of aluminium(III), it is not possible to suggest definite molecular

structures. However, the above studies indicate the presence of a tetra-coordinated aluminium atom in all these derivatives, as shown in Fig. 4-5

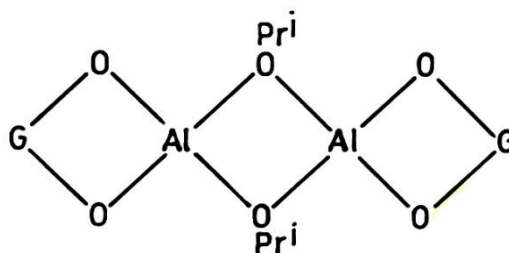


Fig. 4 : Structure of $[(Pr^iO)Al(O-G-O)]_2$

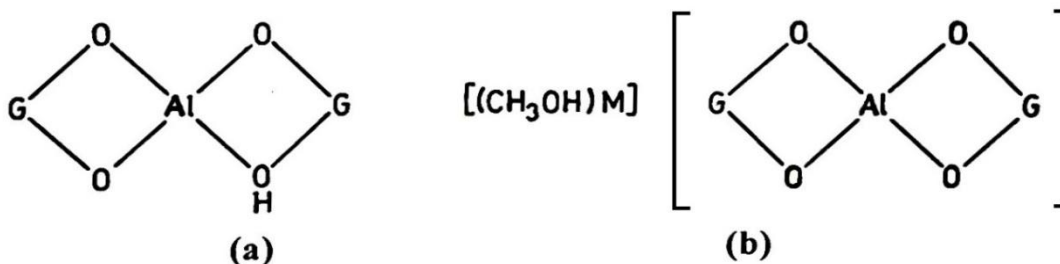
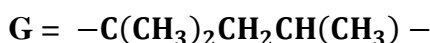
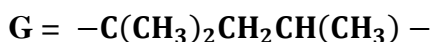


Fig. 5 : (a) Structure of $H[Al(O-G-O)]_2$

(b) Structure of $[(CH_3OH)M][Al(O-G-O)]_2$



References

1. W. D. Treadwell, G. Szabados and E. Haimann. *Helv. Chim. Acta*, 15, 1049-1052 (1932); *CA*, 27, 475 (1933).
2. R. C. Mehrotra and R. K. Mehrotra, *J. Ind. Chem. Soc.*, 39(9), 635-640 (1962).
3. P. Maleki and M. J. Schwing-Weill, *J. Inorg. Nucl. Chem.*, 37, 435-441 (1975); 38, 1787-1788 (1976).
4. R. Benn, A. Rufinska, H. Lehmkuhl, E. Janssen and C. Krüger, *Angew. Chem. Int. Ed. Engl.*, 22(10), 779-780 (1983).
5. M. C. Cruickshank and L.S.D. Glasser, *J. Chem. Soc., Chem. Commun.*, 84-85 (1985).

6. G. J. Gainsford, T. Kemmitt and N. B. Milestone, *Inorg. Chem.*, 34, 5244-5251 (1995).
7. A. I. Vogel, 'Practical Organic Chemistry', Longmans Green, London p. 644 (1948).
8. R. C. Mehrotra, *J. Indian Chem. Soc.*, 30, 585. (1953)
9. D. J. Phillips and S. Y. Tyree, *J. Am. Chem. Soc.*, 83, 1806 (1961).
10. A. K. Sen Gupta, R. Bohra and R. C. Mehrotra, *Inorg. Chim. Acta*, 170, 191 (1990).
11. A. K. Sen Gupta, R. Bohra and R. C. Mehrotra, *Synth. React. Inorg. Metal-Org. Chem.*, 21(3), 445-455 (1991).
12. S. Bhargava, Ph.D. Thesis, University of Rajasthan, Jaipur (1992).
13. A. Singh, A. K. Rai and R. C. Mehrotra, *Indian J. Chem.*, 11, 478-480 (1973).
14. J. H. Wengrovius, M. F. Garbaskas, E. A. Williams, R. C. Going, P. E. Donahue and J. F. Smith, *J. Am. Chem. Soc.*, 108, 982-989 (1986).
15. R. Bohra, A. Dhammani, S. Nagar and R. C. Mehrotra, *Advances in Metallo-Organic Chemistry*, 202-227 (1999).
16. S. Nagar, A. Dhammani, R. Bohra and R. C. Mehrotra, *J. Coord. Chem.*, 55(4), 381-392 (2001).
17. S. Maybodi, Abdolraouf, N. Goudarzi, and H. N. Manesh. *Bulletin of the Chemical Society of Japan* 79(2), 276-281, (2006).