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HETEROCYCLIC COMPOUNDS OF ALUMINIUM (III) WITH GLYCOLS: PART 2 - REACTION OF $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ WITH $MOCH_3$ (M= Li, Na, K) IN 1:3 MOLAR RATIO

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Abstract

Reaction of $Al(OPr^i)_3$ with $HOC(CH_3)_3CH_2CH(CH_3)OH$ in 1:3 molar ratio in refluxing benzene, have resulted in the synthesis of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$. This is soluble in a variety of organic solvents (e.g. benzene, chloroform and dimethylsulfoxide. $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ is monomeric in chloroform. Reaction of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ with $MOCH_3$ (M = Li, Na and K) in 1:3 molar ratio in refluxing methanol yields $[(CH_3OH)M]_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$. 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(^{1}H, ^{13}C and ^{27}Al) spectral studies. ^{27}Al NMR spectra show the presence of four coordinated aluminum site.

Introduction

Aluminium, being a hard acid, is expected to bond strongly to hard bases[1] such as oxygen and/or nitrogen containing ligands. In view of its lesser bonding tendency towards soft bases such as sulphur containing ligands, only a few aluminium derivatives with sulphur-containing ligands have been reported[2-5].

The facile reactivity[6] of metal-alkoxy bond in metal alkoxides with a variety of reagents has been utilized for the synthesis of a number of derivatives for example β-diketonaies[7], carboxylates[8], silyloxides and even heterometallic alkoxide[9]. The feasibility of carrying out such reactions[10-12] in the desired molar ratio with continuous fractionation of alcohol liberated in the reaction azeotropically with benzene has resulted in interesting mixed-alkoxy ligand derivatives.

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In this paper we report the reaction of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ with $MOCH_3$ (M = Li, Na and K) in 1:3 molar ratio.

Experimental

Moisture was carefully excluded throughout experiment. Aluminium isopropoxide was prepared as described by Mehrotra[13]. 2-methyl-2,4-pentanediol was distilled before use. Solvents were dried by reported methods[14].

Aluminium was estimated gravimetrically as the oxinate. Isopropanol was estimated by chromate oxidimetric methods[15]. Isopropoxy groups in the products were estimated by hydrolysing them and collecting the liberated isopropanol azeotropically with benzene.

^1H NMR spectra were recorded using TMS as an internal reference, while ^{13}C and ^{27}Al NMR spectra were recorded in benzene solution using D_2O locks. IR spectra were recorded as Nujol mulls using KBr and CsI plates in the range 4000-200 cm^{-1} on a Perkin-Elmer spectrophotometer model 577. Molecular weight measurements were carried on a Knauer Vapour Pressure Osmometer in chloroform at 45°C.

1. **Reaction of $Al(OPr^i)_3$, with $HOC(CH_3)_2CH_2CH(CH_3)OH$ in 1:3 molar ratio in benzene :** The reaction of $Al(OPr^i)_3$, (3.23 g, 15.81 mmol) with $HOC(CH_3)_2CH_2CH(CH_3)OH$ (5.62 g, 47.55 mmol) in 1:3 molar ratio in refluxing benzene for about 5 hrs. (Completion of the reaction was checked by estimating the isopropanol collected azeotropically with benzene) yielded a colourless viscous liquid of the type $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ after the removal of solvent under vacuum. The product solidified on keeping to give white solid. Yield found : 90%; Anal. found : Al, 7.12; C, 56.85; H, 10.05%. Calculated for $C_{18}H_{39}O_6Al$: Al, 7.13; C, 57.12; H, 10.39%.
2. **Reaction of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ with $LiOCH_3$ in 1:3 molar ratio in methanol :** A methanol solution of lithium methoxide (Prepared by dissolution of 0.20g lithium, 28.82 mmol in excess methanol) was added to a suspension of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ (3.67 g, 9.70 mmol). The mixture was refluxed for 2hrs. to ensure the completion of the reaction. Excess methanol was removed under vacuum, giving a white solid of type $[(CH_3OH)Li]_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$. The product can be

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purified from methanol solution. Yield found : 95% Anal. found : Al, 5.47; C, 50.80; H, 9.46% Calculated for $C_{21}H_{48}O_9AlLi_3$: Al, 5.48; C, 51.22; H, 9.83%.

Likewise, the reactions of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ with sodium or potassium methoxide in 1:3 molar ratio in methanol were carried out (Table-1).

Results and Discussion

Compounds of 2-methyl-2,4-pentanediol

The reaction of $Al(OPr^i)_3$, with $HOC(CH_3)_2CH_2CH(CH_3)OH$ in 1:3 molar ratio in refluxing benzene yield product of the following type :

$$Al(OPr^i)_3 + 3\ HOC(CH_3)_2CH_2CH(CH_3)OH \xrightarrow[\text{Reflux}]{\text{Benzene}} H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3] + 3\ Pr^iOH\ \uparrow$$

This replacement reactions is straight forward up to the liberation of two moles of the isopropanol after that it becomes comparatively slow and are pushed to completion by continuously removing the liberated Isopropanol azeotropically.

This derivatives is highly soluble in benzene, chloroform and dimethyl sulfoxide. $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ is a colourless viscous liquid which undergo solidification on aging. It is momomeric in chloroform.

Reactions of $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ with MOCH in 1:3 molar ratio, in refluxing methanol yields products of the following type:

$$H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3] + 3MOCH_3 \xrightarrow[\text{Reflux}]{\text{Methanol}} [(CH_3OH)M]_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$$

(M = Li, Na and K)

These derivatives are white to brown solids, soluble in methanol and dimethyl sulfoxide, exhibiting high molar conductance in 0.001 M methanol solution[16] (Table-2).

IR Spectra

The IR spectral data for these bimetallic heterocyclic derivatives and the free ligand have been summarized in Table-3. The appearance of a broad band at 3385 cm^{-1} in $H_3[Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3]$ can be ascribed to the –OH group coordinated to aluminium. Presence of a broad band in the IR spectra of other derivatives in the region 3385-3400 cm^{-1} may be assigned to the –OH group of methanol molecule.

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Table 1 : Reaction of $Al(OPr^i)_3$, with $HOC(CH_3)_2CH_2CH(CH_3)OH$ in 1:3 molar ratio and reaction of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ with $MOCH_3$ (M=Li, Na and K)

S. No.	Reactants (g)		Molar Ratio	Product	Pr^iOH (g) found (calcd.)	Yield %	Analysis % found (calcd.)		
	a	b					Al	C	H
1.	$Al(OPr^i)_3$ 3.23	$HOC(CH_3)_2CH_2CH(CH_3)OH$ 5.62	1:3	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	2.84 (2.86)	90	7.12 (7.13)	56.85 (57.12)	10.05 (10.39)
2.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ 3.67	$LiOCH_3$ 0.20	1:3	$[(CH_3OH)Li]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	-	95	5.47 (5.48)	50.80 (51.22)	9.46 (9.83)
3.	3.43	$NaOCH_3$ 0.62	1:3	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	-	98	4.95 (4.99)	46.02 (46.66)	8.90 (8.95)
4.	2.35	$KOCH_3$ 0.73	1:3	$[(CH_3OH)K]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	-	97	4.55 (4.58)	42.17 (42.83)	8.26 (8.22)

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Table 2 : Properties of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ and $[(CH_3OH)M]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ (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.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	Colourless viscous liquid	-	374 (379)
2.	$[(CH_3OH)Li]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	White solid	193	403 (492)
3.	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	Pale yellow solid	196	455 (541)
4.	$[(CH_3OH)K]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	Brown solid	198	500 (589)

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Table 3 : IR spectral data (cm^{-1}) of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ and $[(CH_3OH)M]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ (M = Li, Na and K).

S.No.	Compound	ν O-H	Glycolic ν C-O	Ring vib.	ν Al-O
1.	Ligand $HOC(CH_3)_2CH_2CH(CH_3)OH$	3366 br	1157m	-	-
2.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	3385 br	1025m	945w	625w
3.	$[(CH_3OH)Li]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	3400 br	1025m	945m	645w
4.	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	3385 br	1046m	960m	640w
5.	$[(CH_3OH)K]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	3400 br	1047m	947m	675w

br = broad, m = medium, w = weak

The medium intensity band in the region 1025-1047 cm^{-1} may be assigned to ν C-O vibration[17-19]. A medium to weak intensity band in the region 625-675 cm^{-1} may tentatively be assigned to Al-O stretching vibration[20].

^{1}H NMR Spectra

^{1}H NMR spectra[21] of these complexes and 2-methyl-2,4-pentanediol were taken in $CDCl_3$ 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 $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ indicating the formation of Al-O bonds. $[(CH_3OH)M]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ show a signal at δ 3.51 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 - 1.29, δ 1.49 - 1.56 and δ 4.20 - 4.25 ppm, respectively.

^{13}C NMR Spectra

The ^{13}C 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 ^{13}C nuclei.

In the ^{13}C NMR spectra of all these derivatives, the methyl, methylene, methine and carbonyl carbons, are observed at δ 23.60 - 33.19, δ 49.02 - 49.79, δ 65.07 - 65.72 and δ 69.02 - 71.29 ppm, respectively.

^{27}Al NMR Spectra

^{27}Al NMR spectra of some of these representative derivatives at 23.79 MHz in benzene are summarized in Table-6.

A persual of Table-6 indicates that ^{27}Al NMR chemical shift values are observed in the range δ +50.40 to +70.05 as a broad hump. This indicates[22] the presence of tetra-coordinated aluminium(III) atom in all these derivatives. (Fig. 1)

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Table 4 : ^{1}H NMR spectral data (δppm) of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ and $[(CH_3OH)M]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ (M = Li, Na and K).

S.No.	Compound	Glycolate moiety				Methanol	
		$-CH_3$	$-CH_2-$	-CH<	-OH	$-CH_3$	-OH
1.	Ligand $HOC(CH_3)_2CH_2CH(CH_3)OH$	1.30, m (9H)	1.54, d(2H)	4.51, m(1H)	4.29, br(2H)	-	-
2.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	1.25,m(27H)	1.56, d(6H)	4.25, m(3H)	3.99, br(3H)	-	-
3.	$[(CH_3OH)Li]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	1.29, m(27H)	1.50, d(6H)	4.20, m(3H)	-	3.37, u	3.51, u
4.	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	1.25, m(27H)	1.49, d(6H)	4.20, m(3H)	-	3.37, u	3.51, u
5.	$[(CH_3OH)K]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	1.23, m(27H)	1.52, d(6H)	4.22, m(3H)	-	3.37, u	3.51, u

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

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Table 5 : ^{13}C NMR spectral data (δppm) of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ and $[(CH_3OH)M]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ (M = Li, Na and K).

S.No.	Compound	Glycolate moiety			
		$-CH_3$	$>CH_2/CH^*{}_3OH$	-CH<	>C<
1.	Ligand $HOC(CH_3)_2CH_2CH(CH_3)OH$	25.41,28.93,32.07	50.44	65.96	71.89
2.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	24.59,27.90,31.91	49.79	65.66	71.29
3.	$[(CH_3OH)Li]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	23.60,27.12,30.72	49.02	65.72	69.02
4.	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	25.00,27.68,32.46	49.62	65.07	70.82
5.	$[(CH_3OH)K]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	25.25,27.81,33.19	49.64	65.48	71.05

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Table No. 6 : ^{27}Al NMR Spectral data (δppm) of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$ and $[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$

S.No.	Compound	Shift (δ)	Assignment
1.	$H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	+50.40	Tetrahedral
2.	$[(CH_3OH)Na]_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$	+70.05	Tetrahedral

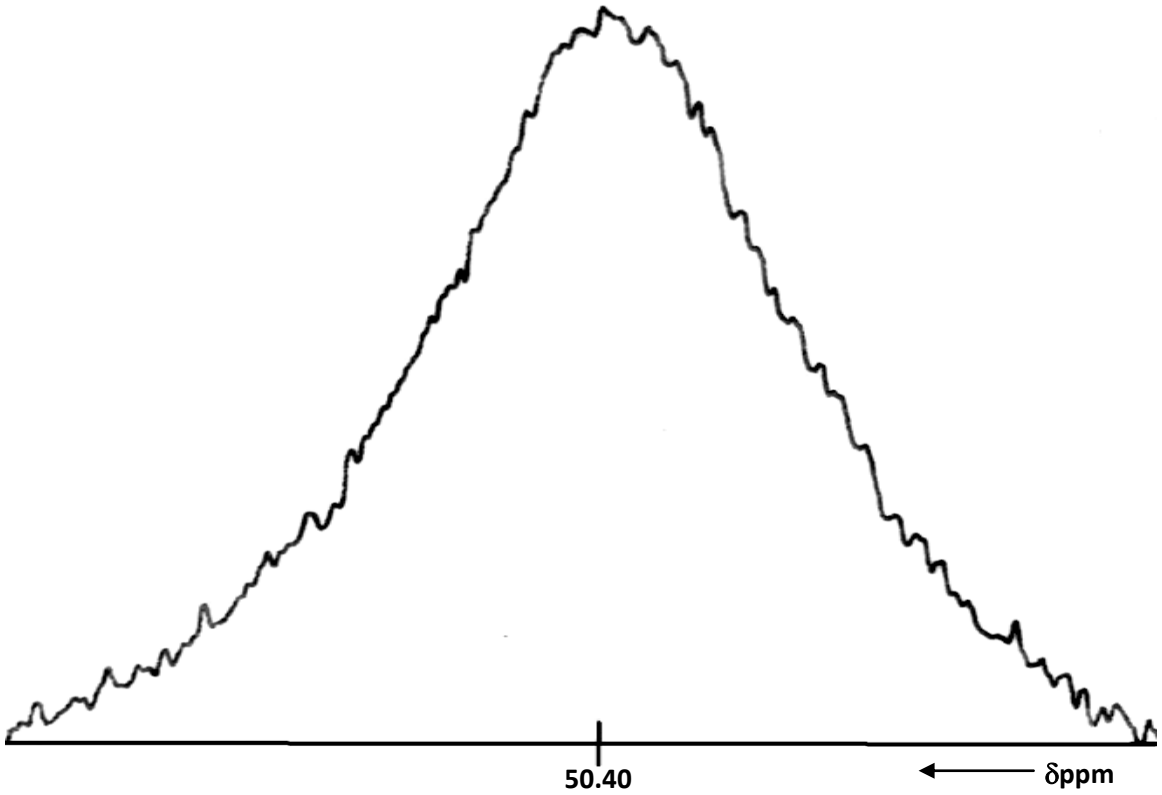

Fig. 1 : ^{27}Al NMR spectrum of $H_3[\overline{Al\{OC(CH_3)_2CH_2CH(CH_3)O\}_3}]$

Structural Features

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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. 2.

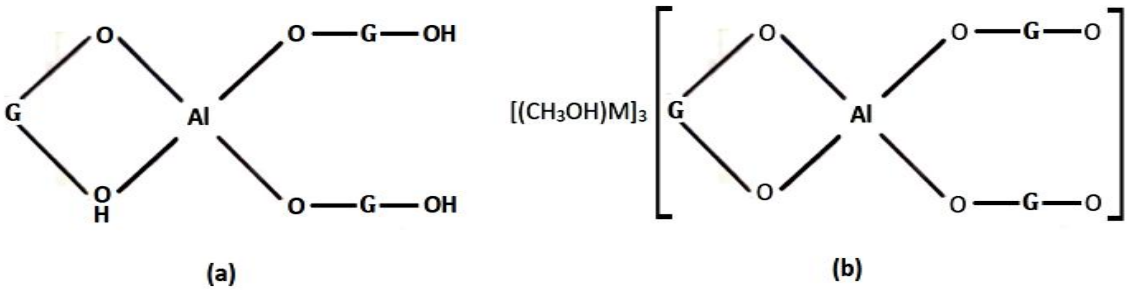

Fig. 2 : (a) Structure of $H_3[\overline{Al(O\text{-}G\text{-}O)_3}]$
(b) Structure of $[(CH_3OH)M]_3[\overline{Al(O\text{-}G\text{-}O)_3}]$
G = $-C(CH_3)_2CH_2CH(CH_3)-$

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