

Synthesis and Characterization of Isomers of Homometallic Dinuclear Aluminium Derivatives of Glycols and Schiff Bases

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ABSTRACT

Isomers of homometallic dinuclear compounds of aluminium with glycols and Schiff bases of the type [(OGO)(OPrⁱ)Al(μ-OPrⁱ)Al[OC₆H₄C(CH₃):NC₆H₄S] and [(OPrⁱ)Al(μ-OPrⁱ)(OGO)Al(OC₆H₄C(CH₃):NC₆H₄S)] where G=MeCHCH₂CMe₂ and Me₂CCMe₂ have been synthesized by the reactions of Al(OPrⁱ)₃ and [(HOGO)AlOC₆H₄C(CH₃):NC₆H₄S] and [OGOAl(HOC₆H₄C(CH₃):NC₆H₄S)] in equimolar ratio, respectively. All these yellow-coloured viscous liquid compounds have been characterized by elemental analysis, molecular weight measurements, and IR and NMR (¹H, ¹³C, and ²⁷Al) spectral data. These aluminium complexes are found to be isomers and have tetra- and hexa-coordinated aluminium atoms.

Key words: Aluminium derivatives, Dinuclear, Glycols, Homometallic, Schiff base.

1. INTRODUCTION

Synthesis and characterization of aluminium complexes with various organic ligands such as Schiff bases, glycols, carbothiamides carboxylates, and diketones have been the area of interest for the researchers due to their diverse and fascinating structural chemistry [1-11]. In these complexes, aluminium has been reported to have tetra, penta, and hexa coordination. Homo and hetero dinuclear compounds of aluminium are also reported in which alkoxy ligands are present as bridging ligands [8-14]. However, there is a scarcity of the dinuclear complexes of aluminium with the isomeric character [12]. In view of the above, some dinuclear aluminium complexes with Schiff base and glycols ligands having isomeric character have been synthesized and characterized during the present investigation and results are being reported in the present paper.

2. MATERIALS AND METHODS

Moisture was carefully excluded throughout the experiments. Al(OPrⁱ)₃ [15] and (OGO)AILH and (HOGO)AIL were prepared by literature methods [12]. Solvents (E. Merck) were dried by standard procedures [16]. Isopropanol in the azeotrope was estimated oxidimetrically [17], using 1N K₂Cr₂O₇ solution in 12.5% H₂SO₄. Aluminium was estimated gravimetrically as oxinate [17]. Nitrogen and sulfur were determined by Kjeldahl and Messenger methods [17], respectively. IR spectra of the ligands and their complexes have been recorded on Nicolet DX FT IR Spectrophotometer using CsI cell. ¹H, ¹³C and ²⁷Al NMR spectra were recorded by Bruker DPX 300 MHz Spectrometer using CDCl₃ solution. Molecular weights of these compounds were determined Ebullioscopically in benzene using Beckmann's thermometer. Since all the reactions have been carried out using similar method, the detail of one representative compound is discussed in detail and the analytical data of the other compounds are summarized in Table 1.

2.1. Synthesis of Compound 3a

The reaction of [(HOGO)AlOC₆H₄C(CH₃):NC₆H₄S] (1.23g, 3.19 mM) was carried out with Al(OPrⁱ)₃ (0.65g, 3.19 mM) in refluxing benzene

(~40 mL) for 24 h and completion of the reaction was checked by estimating the azeotropically liberated isopropanol. The excess solvent of the greenish-yellow colored solution thus obtained was removed under reduced pressure. The compound so obtained was further purified by dissolving it in chloroform and then slowly adding n-hexane until a viscous mass began to settle. The mixture was kept overnight at -5°C. The solution was decanted off and the viscous compound was dried under reduced pressure.

2.2. Synthetic and Analytical Data of the Complexes

2.2.1. Complex 3a

Yield 82%; greenish-yellow viscous; Anal. Calcd. for C₂₆H₃₅NO₅SAI₂ (% Al, 10.19, S, 6.05; N, 2.64 PrⁱOH 0.28 found PrⁱOH Al, 10.14; S, 6.01, N, 2.61 PrⁱOH 0.26 IR (KBr pellets, cm⁻¹): 1458 (ν_{C=C}), 1612 (ν_{C=N}), 1308 (ν_{C-O}), 1015 (ν_{C-O} isopropoxy moiety), 392 (ν_{Al-S}), 525 (ν_{AC-N}), 645-708 (ν_{Al-O}), 735 (ν_{Al-O-Al}), ¹H NMR (CDCl₃, δ_H): 2.38 (s, C(R)=N), 6.49-7.84 (m, 8H Schiff base moiety), 1.14 (s, 64, CMe₂ glycolate moiety), 4.29 (m, ¹H, CHCH₃), 1.36 (d, 3H, CHCH₃) 1.50 (d, 2H, glycolate), 1.14-1.35 (d) 12H, Isopropoxy moiety), 3.74-4.75 (M, 2H, Isopropoxy moiety) ppm ¹³C NMR (CDCl₃, δ_C): 18.35 (s, CH₃), 175.16 (s, EO) 169.55 (s, C=N), 117.27-149.24 (m, aromatic ring). Glycolate moiety: 25.77-26.41 (s, CH₃), 58.62 (s, CH₂), 66.11 (s, OCH) 76.11 (s, >C-O), Isopropoxy moiety 24.32-26.99 (s, CH₃), 65.19-67.0 (s, OCH) ppm. ²⁷Al NMR (C₆H₆, δ ²⁷Al) 60.1, 1.82 ppm MW 525 Calc. 529.6.

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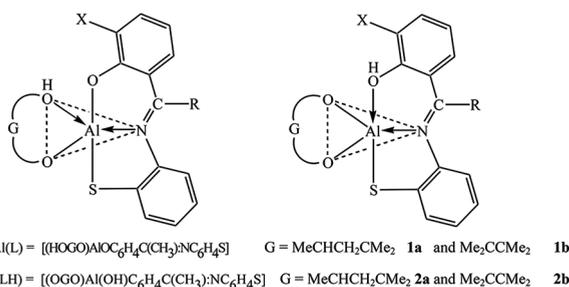
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2.2.2. Complex-3b

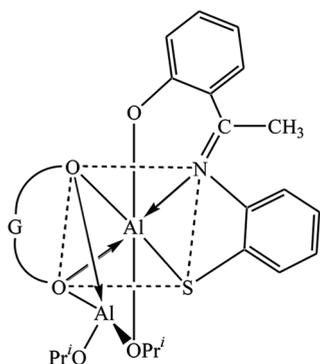
Yield 86%; yellow semisolid, Anal., Calcd. for $C_{26}H_{35}NO_5SAI_2$ (%) Al, 10.19; s, 6.05, N, 2.64 Pr^iOH , 0.31) found Al, 10.13; S, 5.99, N 2.60; Pr^iOH 0.30, IR (KBr pallets, cm^{-1}) Schiff base moiety: 1460 ($\nu_{C=N}$), 1305 (ν_{C-O}); Isopropoxy moiety: 1018 (ν_{C-O}); 395 (ν_{Al-S}), 550 (ν_{Al-N}), 640-700 (ν_{Al-O}), 730 ($\nu_{Al-O-Al}$) 1H NMR ($CDCl_3$, δ H): Schiff base moiety: 2.45 (s, C(R)=N 6.37-7.83 (M aromatic ring), Glycolate moiety 1.17 (s, -CMe₂); Isopropoxy moiety 1.15 (6H, Al-OPrⁱ) 1.32 (6H Al-OPrⁱ-Al), 3.58 (M, 1H , Al-OPrⁱ), 4.31 (M, Al-OPrⁱ-Al), ^{13}C NMR ($CDCl_3$, $\delta^{13}C$) 18.36 (s, CH₃), 175.15 (s, CO), 168.69 (s, C=N) 118.28-149.39 (s, aromatic ring).

2.2.3. Complex 4a

Yield 84% yellow semisolid Analytical data: Calculated for $C_{26}H_{35}NO_5SAI_2$ Al 10.19, s 6.05, N 2.64, Pr^iOH 0.30, found Al 10.15, s 6.01, N 2.59 Pr^iOH

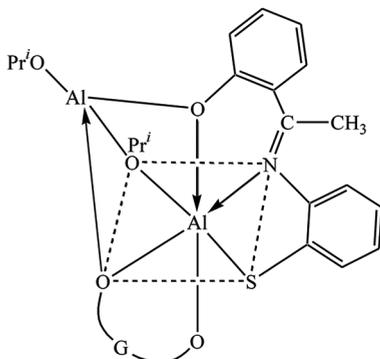


Scheme 1: Synthesis of homometallic dinuclear compounds of aluminium.



G = (CH₃)CH₂CH(CH₃) (**3a**) and (CH₃)₂C-C(CH₃)₂ (**3b**)

Figure 1: Structure of [OGO(OPrⁱ)Al(μ -OPrⁱ)AlOC₆H₄C(CH₃):NC₆H₄S].



G = (CH₃)CH₂CH₂CH(CH₃) (**4a**) and (CH₃)₂C-C(CH₃)₂ (**4b**)

Figure 2: Structure of [SC₆H₄N: C(CH₃)C₆H₄OAlOGO(μ -OPrⁱ)Al(OPrⁱ)].

0.28, IR (KBr pallets, cm^{-1}) Schiff base moiety: 1465 ($\nu_{C=C}$), 1620 ($\nu_{C=N}$), 1300 (ν_{C-O}), Isopropoxy moiety 1020 (ν_{C-O}), 410 (ν_{Al-S}), 585 (ν_{Al-N}), 625-698 (ν_{Al-O}) 745 ($\nu_{Al-O-Al}$), 1H NMR ($CDCl_3$, δ H): Schiff base moiety 2.61 (s, (R)=N), 6.47-7.80 (aromatic ring), Glycolate moiety 1.21 (s, -CMe₂), 4.17 (M, 1H), 1.35 (d, 3H) 1.84 (s, 2H), Isopropoxy moiety 1.21 (6d AlOPrⁱ) 1.42 (64d, Al-OPrⁱ-Al), 3.58 (M, 1H Al-OPrⁱ) 4.37 (M, 1H Al-OPrⁱ-Al). ^{13}C NMR ($CDCl_3$, $\delta^{13}C$) 175.12 (s, CO), 118.28-149.39 (s, Aromatic ring), Glycolate moiety 25.91-26.90 (s, CH₃), 55.7 (s, 2H) 64.9 (s, OCH) 74.1 (s, CO), Isopropoxy moiety 24.42-26.98 (s, CH₃) 65.70-66.90 (s, OCH), ^{27}Al nmr (C₆H₆, $\delta^{27}Al$) 65.1, 1.95 mw 524 Cald. 529.6.

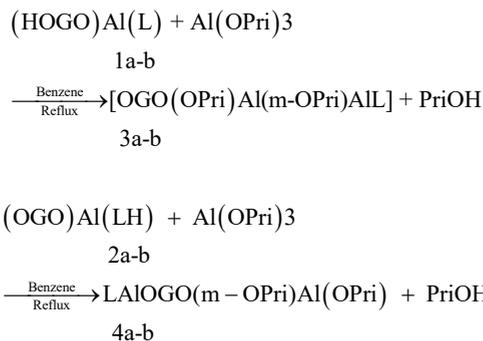
2.2.4. Complex 4b

Yield 83% yellow semi-solid, Analytical data: Calculated for $C_{26}H_{35}NO_5SAI_2$ Al, 10.19, s, 6.05, N, 2.64, Pr^iOH , 0.32; found Al, 10.13, s, 6.05, N, 2.64, Pr^iOH , 0.32 IR (KBr pallets, cm^{-1}) Schiff base moiety: 145 ($\nu_{C=C}$), 1625 ($\nu_{C=N}$), 1307 (ν_{C-O}), Isopropoxy moiety 1016 (ν_{CO}), 610 (ν_{Al-N}), 635, 725 (ν_{Al-O}), 750 ($\nu_{Al-O-Al}$), 1H NMR ($CDCl_3$, δ H): Schiff base moiety 2.57 (s, 3H), 6.34-7.76 (M, aromatic ring), Glycolate moiety: 1.18 (s, 6H, CMe₂), Isopropoxy moiety 1.18 (d6H, terminal OPrⁱ), 1.43 (d, 6H bridging OPrⁱ), 3.49 (M, 1H , terminal OPrⁱ), 4.69 (M, 1H , bridging OPrⁱ). ^{13}C NMR ($CDCl_3$, $\delta^{13}C$) Schiff base moiety: 18.32 (s, CH₃), 175.1 (s, CO), 168.09 (s, C=N), 114.68-149.52 (Aromatic ring), Glycolate moiety 24.83-25.24 (s, CH₃) 74.65-74.98 (>CO) Isopropoxy moiety 24.83-27.55 (s, CH₃), 65.57-67.75 (s, OCH), ^{27}Al (C₆H₆, $\delta^{27}Al$) 51.0 (hexa), 2.3 (tetra).

3. RESULTS AND DISCUSSION

3.1. Synthesis

Reactions of the our earlier reported aluminium derivatives [(HOGO)AlOC₆H₄C(CH₃):NC₆H₄S] and [(OGO)Al(OH)C₆H₄C(CH₃):NC₆H₄S] [12] were carried out with Al(OPri)₃ in 1:1 molar ratios to yield the dinuclear aluminium complexes [(OGO)(OPri)Al(μ -OPri)AlOC₆H₄C(CH₃):NC₆H₄S] and [SC₆H₄N: C(CH₃)C₆H₄OAlOGO(μ -OPri)Al(OPri)], respectively, as per the scheme 1 given below-



The reaction mixture was refluxed on a fractionating column for ~ 4 h. The isopropanol thus liberated was fractionated off azeotropically with benzene. The excess solvent was removed under reduced pressure. These yellow colored viscous compounds are soluble in common organic solvents and found to be monomeric in refluxing benzene solution.

3.2. Spectroscopic Studies

A comparison of IR spectra of these newly synthesized aluminium complexes (3a, 3b, 4a, and 4b) with the corresponding precursors [1a, 1b, 2a, and 2b] shows the disappearance of glycolic OH (1a, 1b) and phenolic -OH (2a, 2b) bands which are observed as broad bands at 3085-3328 cm^{-1} and 3271-3457 cm^{-1} in the spectra of corresponding aluminium derivatives (HOGOAIL) and (OGOAILH), respectively. This indicates the formation of a new Al-O bond in these complexes. The appearance of a new band in the region 625-725 cm^{-1} for vAl-O [2,13] further confirms the formation of this bond.

Table 1: Synthetic and analytical data of isomers of dinuclear aluminium complexes.

S. No	Complex	Reactants g (mM)		Molecular formula	yield %	Pr ⁱ OH found (Calc.) g	% Elemental analysis found (Calc.)			Molecular weight found (Calc.)
		Precursor	Al (OPr ⁱ) ₃				Al	S	N	
1.	OGO (OPr ⁱ) Al(μOPr ⁱ) AlOC ₆ H ₄ C (CH ₃):NC ₆ H ₄ S G = -CHMeCH ₂ CMe ₂ -	1.77 (4.59)	0.94 (4.60)	C ₂₆ H ₃₅ NO ₅ Al ₂ 82		0.26 (0.28)	10.14 (10.19)	6.01 (6.05)	2.61 (2.64)	525 (529.6)
2.	OGO (OPr ⁱ) Al(μOPr ⁱ) AlOC ₆ H ₄ C (CH ₃):NC ₆ H ₄ S G = -CMe ₂ CMe ₂ -	1.98 (5.14)	1.05 (5.14)	C ₂₆ H ₃₅ NO ₅ Al ₂ 86		0.30 (0.31)	10.13 (10.19)	5.99 (6.05)	2.60 (2.64)	524 (529.6)
3.	SC ₆ H ₄ N: C (CH ₃) C ₆ H ₄ OAlOGO(μ-OPr ⁱ) Al (OPr ⁱ) G = -CHMeCH ₂ CMe ₂ -	1.92 (4.98)	1.02 (4.99)	C ₂₆ H ₃₅ NO ₅ Al ₂ 84		0.28 (0.30)	10.15 (10.19)	6.01 (6.05)	2.59 (2.64)	524 (529.6)
4.	SC ₆ H ₄ N: C (CH ₃) C ₆ H ₄ OAlOGO(μ-OPr ⁱ) Al (OPr ⁱ) G = -CMe ₂ CMe ₂ -	2.05 (5.32)	1.09 (5.34)	C ₂₆ H ₃₅ NO ₅ Al ₂ 83		0.30 (0.32)	10.13 (10.19)	5.98 (6.05)	2.60 (2.64)	523 (529.6)

Table 2: ¹H and ²⁷Al NMR spectral data (δ ppm) for the isomers of dinuclear aluminium compounds.

Compound	Schiff base moiety		Glycolate moiety			Isopropoxy moiety		²⁷ Al
	C (R)=N	Aromatic proton	-C (CH ₃) ₂	-CH (CH ₃)	-CH ₂	CH ₃	OCH	
3a	2.38	6.49–7.84	1.14	4.29(m, 1H) 1.36(d, 3H)	1.50 (d, 2H)	1.14 1.35	3.74 4.75	60.1 1.82
3a	2.45	6.47–7.83	1.17 (s, 12H)	-	-	1.15 1.32	3.58 4.31	59.6 1.72
4a	2.61	6.47–7.80	1.21	4.17(m, 1H) 1.35(d, 3H)	1.84 (d, 2H)	1.21 1.42	3.58 4.37	65.1 1.95
4b	2.57	6.34–7.76	1.18	-	-	1.18 1.43	3.49 4.69	51.0 2.3

Table 3: ¹³C NMR Spectral data (δ ppm) of isomers of dinuclear aluminium compounds.

Compounds	Schiff base moiety				Glycolate moiety				Isopropoxy moiety	
	CH ₃	C-O	C=N	Aromatic proton	-CH ₃	-CH ₂	-OCH	>C-O	CH ₃	OCH
1	18.35	175.16	169.55	117.27–149.25	25.77 25.92 26.41	58.62	66.11	76.11	24.32 26.99	65.19 67.07
2	18.36	175.15	168.69	118.28–149.39	24.92 24.78	-	-	74.78 74.99	24.65 27.01	65.25 66.88
3	18.33	175.12	168.12	118.28–149.39	25.91 25.97 26.90	55.7	69.9	74.1	24.42 26.98	65.70 66.99
4	18.32	175.17	168.09	114.68–149.52	24.83 25.24	-	-	74.65 74.98	24.83 27.55	65.57 67.75

A medium-intensity band observed at 1015–1020 cm⁻¹ is assigned to νC-O of the isopropoxy group [14]. The Al-O-Al vibrations are observed at 730–750 cm⁻¹ [13] indicating the formation of dinuclear aluminium compounds. The absorption band for phenolic C-O is observed with a shift in its position as compared to the precursor of the complexes indicating that the phenolic oxygen of Schiff base is involved in coordination [2,13]. Absorption bands for νC=N, νAl←N and νAl-S are observed at 1612–1632 cm⁻¹, 525–610 cm⁻¹, and 392–442 cm⁻¹, respectively.

A comparison of ¹H NMR spectra [Table 2] of these newly synthesized 3a and 3b aluminium complexes with the corresponding precursors 1a and 1b shows the absence of glycolic -OH. This signal is observed at δ 4.17–

4.46 ppm in the spectrum of (HOGAIL) [12]. Likewise, the phenolic OH signal also disappeared on further complexation of 2a and 2b to result the complexes 4a and 4b. The phenolic -OH signal is observed in the range δ 12.48–13.09 ppm in the spectra of OGOAILH [12].

The presence of two doublets (at δ 1.14–1.21 and 1.32–1.43 ppm) and two multiplets (at δ 3.49–3.74 and 4.31–4.75 ppm) for each methyl and methine protons, respectively, indicates the presence of bridging and terminal isopropoxy groups. A singlet observed at δ 2.38–2.61 ppm has been assigned to C(CH₃)=N protons of Schiff's base moiety. The Schiff base aromatic ring protons are observed as multiplet in the range δ 6.34–7.84 ppm. Protons of glycolic moiety are observed with small downfield shifts at their expected positions.

A comparison of ^{13}C NMR spectra [Table 3] of these newly synthesized aluminium complexes with their precursors shows the appearance of some new signals. The two carbon signals observed in the range δ 24.32–24.83 and 26.98–27.55 ppm and two more signals in the range δ 65.19–65.70 and δ 66.88–67.75 ppm for gem dimethyl and methine carbons of isopropoxy group in the complexes indicates the presence of bridging and terminal isopropoxy groups [13] in these derivatives. ^{13}C NMR spectra of all these derivatives show a downfield shift in the position of phenolic C-O signals as compared to their position in the corresponding precursor. The signal for C=N carbon appears at δ 168.09–169.55 ppm in these complexes confirming the formation of $>\text{C}=\text{N}\rightarrow\text{Al}$ bond. The glycolate carbon signals are observed at their expected positions and numbers.

^{27}Al NMR spectra of these compounds recorded at room temperature exhibit two signals at δ 1.72–2.3 ppm and δ 51.0–65.1 ppm which indicates the presence of hexa- and tetra-coordinated aluminium atoms in these complexes [1,13].

As mentioned above, these complexes are monomeric and have the presence of bifunctional tridentate Schiff base and bifunctional glycolate moiety. The present studies also confirm the presence of bridging and terminal isopropoxy groups in these complexes. The ^{27}Al NMR spectral data indicate hexa- and tetra-coordination around central aluminium atoms in these complexes. Spectral studies also indicate that there is replacement of glycolic proton by $\text{Al}(\text{OPr}^i)_2$ in complexes 3a and 3b. Likewise, there is replacement of phenolic proton by $\text{Al}(\text{OPr}^i)_2$ in the complexes 4a and 4b. It means that complexes 3a and 3b are isomers of the complexes 4a and 4b, respectively.

In view of the above, following structures [Figures 1 and 2] may be proposed for these homometallic dinuclear isomeric complexes.

4. ACKNOWLEDGMENT

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5. CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this article.

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*Bibliographical Sketch



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