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Simple generation of neutral bimetallic aluminium and zinc alkyls Schiff bases bridged by a central resorcinol moiety

Research Article

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Abstract: Aluminium and zinc complexes bearing the N,O-chelating Schiff base ligand 4,6-bis-1-(2-(dimethylamino)ethylimino)ethyl)benzene-1,3-diol, (C_eH₂(OH)₂(NCH₂CH₂NMe₂)₂) (1a), have been synthesized. Bimetallic aluminium and zinc alkyl complexes (2a - 4a) were prepared by treatment of the hexadentate 1a with the appropriate amount of AlMe₃, ZnMe₂ and ZnEt₂, respectively. 2a has been characterized crystallographically, it lies on a crystallographic two-fold rotation axis and each aluminium centre adopts a five coordinate geometry. Complex 2a was tested as a catalyst in the ring-opening polymerisation of ε-caprolactone. We describe here the synthesis of two neutral ligands (1a and (C_eH₂(OH)₂(C_eH₅NH₂) (C=O(CH₃)) (1b)) and demonstrate their application in the synthesis of molecular aluminium and zinc derivatives.

Keywords: Aluminium methyl • Zinc alkyl • Schiff bases • DAR • Binuclear complex

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1. Introduction

Schiff base ligands are easily synthesized and form complexes with almost all metal ions, many of their complexes show high catalytic activity [1]. Monometallic Al complexes containing a series of phenoxy-imine ligands or NN Schiff base ligands have been reported [2-11]. The monometallic pendant-arm single Schiff base complexes of aluminium provide active centers for ethylene polymerization due to the liability of the pendant donor arm, thus allowing a pathway for ethylene to approach the aluminium centre [6]. So far, considerable attention has been paid to the synthesis, structural determinations, and catalytic activity of metal complexes based on aluminium and zinc [12-15]. Among the reported catalysts, bimetallic complexes are relatively few [16-19], and those reported for binuclear zinc alkyl, aryl and aryloxide involved Cl, N and O bridging ligands [20-22].

The bifunctional carbonyl compound 4,6-diacetylresorcinol (DAR) serves as precursor for the

generation of symmetrical Schiff bases which are either di- or tetra-basic with two symmetrical sets of either O_2N or N_2O tridentate chelating sites [23-27]. Nevertheless, dinuclear Al(III) and Zn(II) alkyl complexes with 1,3-dihydroxybenzene bridging motifs are lacking.

Here we have synthesized the new double and single-Schiff-base ligands ${\bf 1a}$ and ${\bf 1b}$, respectively, as shown in Scheme 1, starting from DAR by Schiff-base condensation with two equivalents of the appropriate amine. The resulting ligand (${\bf 1a}$) provides hexadentate [N₂O] binding pockets which are bridged by a central resorcinol moiety, while (${\bf 1b}$) provides both tridentate N₂O and bidentate OO chelating sites.

2. Experimental Procedure

2.1. Chemicals and physical measurement

All manipulations were carried out in an atmosphere of dry nitrogen using standard Schlenk techniques or in an inert-atmosphere glovebox. Solvents were dried from the appropriate drying agent, distilled, degassed



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