

Synthesis and molecular structures of the magnesium and aluminum adducts of a niobium-oxo complex. X-ray crystal structures of  $[\{\text{NbOCl}_4(\text{THF})\}_2\text{Mg}(\text{THF})_4]$  and  $[\{\text{NbOCl}_4(\text{THF})\}_2\text{AlCl}(\text{THF})_3]$ .

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Streszczenie

The most active Ziegler–Natta catalysts of alkene polymerization are  $d^0$  metallocenes of the XIII, XIV, and XV groups.<sup>1</sup> On the other hand, vanadium catalysts<sup>2</sup> generally show a  $d^2$  or  $d^3$  configuration. However,  $d^0$  vanadium complexes have frequently been used as catalysts but were readily reduced to a lower oxidation state in the presence of a cocatalyst. The synthesis of high polymers based on simple Nb(V) species as catalysts has also been reported.<sup>3</sup> It could be expected that V(V) and Nb(V) complexes would be effective catalysts if they could be stabilized in their high oxidation state.<sup>4</sup> In this contribution, we describe the synthesis and characterization of  $\text{Mg}^{2+}$  and  $\text{Al}^{3+}$  niobium–oxo adducts. The chemistry of transition metal–oxo  $\text{M}=\text{O}$  (V, Nb, Ta) adducts with electropositive metal ions such as  $\text{Mg}^{2+}$  or  $\text{Al}^{3+}$  as components of the catalyst is unknown yet. In a previous paper we described the synthesis and properties of products formed during reaction between  $[\text{MoOCl}_3(\text{THF})_2]$  and  $[\text{MgCl}_2(\text{THF})_2]$ .<sup>5</sup> For a Mo/Mg 2:1 molar ratio in THF a crystalline salt  $[\text{Mg}(\text{THF})_6][\text{MoOCl}_4(\text{THF})_2]$  is formed which reacts further with 3 equiv of bis(tetrahydrofuran)magnesium dichloride yielding the ionic  $[\text{Mg}_2(\mu\text{-Cl})_3(\text{THF})_6][\text{MoOCl}_4(\text{THF})]$  compound. The  $[\{\text{MoOCl}_4(\text{THF})\}_2\text{Mg}(\text{THF})_4]$  and  $[\text{MgMo}(\mu\text{-Cl})_3\text{Cl}_2\text{O}(\text{THF})_3]$  molecular compounds were readily obtained by interaction of  $[\text{MoOCl}_3(\text{THF})_2]$  with  $[\text{MgCl}_2(\text{THF})_2]$  in a 2:1 and 1:1 molar ratio in  $\text{CH}_2\text{Cl}_2$ , respectively. However, compounds  $[(\text{ReMe}_4\text{O})_2\text{Mg}(\text{THF})_4]$  and  $[\{o\text{-C}_6\text{H}_4(\text{CH}_2)_2\text{WO}\}_2\text{Mg}(\text{THF})_4]$  arise from Grignard reagents or  $\text{MgR}_2$  with  $\text{R}_2\text{O}_7$  or  $\text{WCl}_4\text{O}$  in THF.<sup>6,7</sup> Similar adducts with  $\text{Al}^{3+}$  were unknown.

Słowa kluczowe

Adducts, Catalysts, Cations, Molecules, Tetrahydrofurans

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