Keywords: silver perfluorocarboxylates; alcohol; single-crystal-to-single-crystal reaction

FA4-MS06-P10

Synthesis and Structural Characterization of New Heteroleptic Aluminium Alkoxides and Siloxides.

Sergio Pedrosa, Marta E.G. Mosquera, Pilar Gómez-Sal. Departamento de Química Inorgánica. Universidad de Alcalá, Campus Universitario, Alcalá de Henares, 28805-Madrid, Spain.

Aluminium attracts wide attention not only because of its rich chemistry, but also due to the important applications that exhibit in areas as diverse as organic synthesis, electronic materials, structural materials and catalysis.[1] In particular, aluminoxanes play a key role as co-catalysts in the Ziegler-Natta polymerization processes.[2] As well, aluminium alkoxide derivatives have proven to be very efficient catalysts in many polymerization reactions, such as ring opening polymerization.[3] As a continuation of our work on functionalized aryloxide aluminium derivatives,[4] we have extended our studies to silanols. The generation of molecular aluminosiloxanes compounds has an additional interest since this kind of species have shown to be useful both as model compounds for open-framework silicates and as precursors for the preparation of mixed metal oxide materials under mild conditions.[5] We are particularly interested in achieving a good control of the structures obtained by controlling the reactions conditions. In this communication we present our latest results and our studies in relation to the different outcome depending on the aluminum precursor. Thus when AlMe₃ is used the expected dimer [AlMe₂(OR)]₂ is formed, however if AlCl₃ is the precursor, unexpected results are attained such as the mixed alkoxide/siloxide derivative [AlCl(OR)OSi(OR)]₂, shown in figure 1.

![Figure 1](image-url)

The new derivatives prepared were determined by X-ray diffraction methods, elemental analysis and NMR.


Keywords: aluminium compounds; alkoxides; inorganic and organometallic compounds

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Double Complex Salts with [Ru(NH₃)₅Cl]²⁺ Cation and [OsCl₆]²⁻ Anion: Synthesis and Properties.


E-mail: x-vizor@yandex.ru

Recently, interest in new energy sources and accumulators based on superfine metal catalysts deposited on the carbon supporter was grown [1, 2]. Ruthenium containing alloys show an extraordinary electrochemical activity and CO resistance [3]. This work continues a research of double complex salts, which are perfect precursors for obtaining solid solutions of platinum metals. [Ru(NH₃)₅Cl][OsCl₆] (I) was obtained by mixing of hot water solutions of [Ru(NH₃)₅Cl]Cl₂ and Na[OsCl₆]. The residue was washed with water and acetone, then dried in air. Yield is 85 - 90%. A synthesis of [Ru(NH₃)₅Cl][OsCl₆]Cl₂ (II) was carried out with mixing of [Ru(NH₃)₅Cl]Cl₂, powder, Na[OsCl₆] water solution and 0,1 M HCl. The mixture was left for 5 days in the dark place. The formed residue was washed with water and acetone, then dried in air. Yield is 85 - 90%. Cell parameters of I were refined by Rietveld method using [Rh(NH₃)₅Cl]Cl₂ structure as a model [5]: a = 11,593(1), b = 8,318(1), c = 15,234(4). β = 90,70(4)°; V = 1469 Å³; space group P2₁/m; Z = 4. The crystal structure of I was determined with a X8APEX Bruker diffractionsystem (MoKα-radiation, graphite monochromator). Crystal structure data: C2/m space group, a = 11,1849(8) Å, b = 7,9528(6) Å, c = 13,4122(9) Å, V = 1175,75 Å³, Z = 2. According to XRD data, II is isostructural to [M(Cl)(NH₃)₅][M₄Cl₄]Cl₂, where M¹ = Rh, Ir, Co and M² = Re, Os, Ir, Pt [4]. Os-Cl bond lengths in the complex anion lay between 2,328-2,356 Å, Ru-N average bond length in complex cation is 2,111 Å, Ru-Cl is 2,334 Å. Distances between metals are typical for this kind of compounds. The smallest Ru…Os distance is 5,773 Å. Thermal stability of the complexes in different atmospheres was also investigated. It was found that the decomposition of I or II in hydrogen or helium atmosphere at 650 °C gives a single-phase metal product with the hcp lattice. Cell parameters of obtained phases are close to cell parameters of original Ru-Os alloys obtained at 2000° C [6, 7].


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