MS61.P13

Discrete role of chlorine substitutions in the conformation and supramolecular architecture of isostructural arylsulfonylamides

William B. Fernandes, a Angelo Q. Aragão, a Felipe T. Martins, a Caridad Noda-Perez, a Carlito Lariucci, a Hamilton B. Napolitano, a *Science and Technology Center, State University of Goiás, Anápolis, (Brazil). b Institute of Chemistry, Federal University of Goiás, Goiânia, (Brazil). c Institute of Physics, Federal University of Goiás, Goiânia, (Brazil). E-mail: willfernandes@upec.ug.br

Compounds containing sulfonyamide groups SO₂NH occur in many biologically active molecules, including antimicrobial, antithyroid, antitumor and antimalarial drugs [1,2,3,4]. In this study, two arylsulfonylamide derivatives differing for two chlorine substitutions at one of their two phenyl rings were synthesized and characterized by X-ray diffraction technique in order to establish structural relationships and the chlorine role in the conformation and crystal assembly. The compounds (4-N[(2, 5-dichlorophenyl)sulfonyl-amide-acetophenone]) (I) and 4-N [(2, 5-dichlorophenyl)sulfonyl-amide-acetophenone] (II) were obtained by the equimolar coupling between benzene sulfonyl chloride or 2,5-dichlorobenzene sulfonyl chloride and 4-amineacetophenone in dichloromethane or acetone as solvent at 343K for until six hours. The precipitate was re-crystallized in suitable solvents to obtain the single crystals. Crystallographic data for compounds (I) and (II) were obtained by the equimolar coupling between benzene sulfonyl chloride or 2,5-dichlorobenzene sulfonyl chloride and 4-amineacetophenone in dichloromethane or acetone as solvent at 343K for until six hours. The precipitate was re-crystallized in suitable solvents to obtain the single crystals. Crystallographic data for compounds (I) and (II) were collected using a Enraf Nonius diffractometer at room temperature. All crystal structures were solved by Direct Methods, and refined by full matrix least square method on F². The non hydrogen atoms were refined anisotropically. All C-H hydrogens were placed geometrically and refined using riding model. The hydrogen H1 bonded to nitrogen was found from the difference Fourier map and its positional parameters were refined freely. The tables, geometrical calculations, molecular packing and drawings were done with the WinGX [5,6] program package, obtaining the following results: (I) Molecular formula: C₂₇H₂₃NO₂S. Unit Cell Parameters: a = 13.0007(5) Å, b = 8.3615(4) Å, c = 12.5179(6) Å, \( \beta = 98.118(3)^\circ \), \( \alpha = 99.180(1)^\circ \), \( \gamma = 134.713(1)^\circ \). 5393 measured reflections with 3009 unique and 2214 observed. Final indices \( R_p = 0.0483 \) for 177 refined parameters. (II) Molecular formula: C₂₇H₂₃NO₂S. Unit Cell Parameters: a = 13.3622(2) Å, b = 8.1542(2) Å, c = 12.0007(5) Å, \( \beta = 98.118(3)^\circ \), monoclinic, space group P2₁/c, Z = 4, \( V = 13477.13(1)^3 \) Å³, 5653 measured reflections with 2972 unique and 2463 observed. Final indices \( R_p = 0.0494 \) for 195 refined parameters. There are several classical and non-classical hydrogen bonds and \( \pi-\pi \) stacking interactions contributing to stabilize the crystal packing of the compounds.

Keywords: aluminate, gallium, titanium

MS61.P12

Incorporation of different lithium aluminates to titanium oxide organometallic ligands

Alberto Hernán-Gómez, Avelino Martín, Miguel Menáa and Cristina Santamaría, Departamento de Química Inorgánica, Universidad de Alcalá. Campus Universitario. 28871-Alcalá de Henares-Madrid (Spain). E-mail: alberto.hernan@uah.es

During the last years our research group has investigated the coordination of the organometallic oxides \([\text{Ti}(\eta^1\text{-C}_6\text{Me}_3)(\mu-O))\{\mu_{3}-\text{CR})\}\) \((R = \text{H}, \text{Me}2)\) to different inorganic fragments of groups 1 and 13 elements. Reactions with group 1 derivatives lead to dehydrogenation processes of the alkylidyne moiety to give [M(\mu_3-O)(\mu-O)\{\text{Ti}(\eta^1\text{-C}_6\text{Me}_3))(\mu_{3}-\text{CR})\}] (M = Li, Na, K) complexes [1]. On the other hand, the novel adducts \([\{\text{E,M}(\mu, O)-O)(\mu-O)\{\text{Ti}(\eta^1\text{-C}_6\text{Me}_3))(\mu_{3}-\text{CR})\}]\) \((R = \text{H}, \text{Me}; M = B, Al, Ga; E = \text{alkyl, haloalkyl, halide})\) are obtained when group 13 derivatives react with 1 and 2 [2]. These species exhibit coordination of 1 or 2 through one of the three oxygen atoms of the TiO₂ ring to the metal center. Now, we are studying the chemical reactivity of this family of compounds with different alkyl, amide or alkoxyde lithium derivatives in order to isolate and synthesize these organometallic oxides in a chelate tridentate fashion. Now, this bonding system is stabilized by the alkyl aluminum fragment, which avoids that the amide or alkoxyde fragments react with the alkylidyne moiety (see figure).

However, when the same reaction is carried out with \( p \)-tolyl lithium and the triphenylgallium adduct, abstraction and redistribution reactions are observed. The lithium atom is then encapsulated by two units of 1 and the new gallate species stabilizes a phenyllithium unit.

Keywords: pyridoxal, thiosemicarbazone, copper