

s13.m38.p3 **Crystallographic and IR-spectroscopic Studies of [Cu(imidazole)₄(NO₂NCN)₂].** Jesús García Díaz^a, Anton Gatial^b, Ingrid Svoboda^c and Jozef Kozisek^b, ^aMorelia Technological Institute, Morelia, Michoacan, Mexico, ^bDepartment of Physical Chemistry, Slovak University of Technology, Bratislava, Slovakia, and ^cMaterials Science, Darmstadt University of Technology, Darmstadt, Germany. E-mail: jesus1250@yahoo.com.mx

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The cyanamidonitrate ligand has potentially four donor atoms: N(amide), N(nitrile) and two O atoms. The variety of coordination opportunities for cyanamidonitrate ligand (monodentate, or bidentate bridging ligand), in dependence on the kind of the central atom, especially on its hardness is expected. New cyanamidonitrate complex of composition [Cu(NO₂NCN)₂(im)₄] (where (im) = imidazole) was prepared and the crystal structure solved. Data collection was performed on Xcalibur diffractometer and the data reductions were done both for the triclinic (a=9.825, b=15.217, c=15.219 Å, α=104.34, β=96.85 and γ=96.67°), as well as monoclinic (a=18.668, b=24.038, c=9.825 Å, β=101.06 °) crystal system. Crystal structure with high R-value was solved only in triclinic system. As the crystal quality is poor, the IR spectra were introduced to distinguish whether there are more than one independent molecule in the unit cell. According to the crystal field splittings it might be expected that at least two independent molecules are in the unit cell. Preliminary results indicate the pseudo-octahedral coordination of the central atom. In equatorial plane Cu(II) is coordinated by four donor nitrogens from imidazole ligands and in the axial position by two nitrile donor nitrogens from cyanamidonitrate anions.

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s13.m38.p4 **A New Barium Biglutarate: Synthesis, X-Ray Structure and Thermal Behavior.** K. Aliouane⁽¹⁾, A. Djeghri⁽¹⁾, A. Guehria-Laidoudi⁽¹⁾, S. Dahaoui⁽²⁾, C. Lecomte⁽²⁾, ⁽¹⁾Laboratoire de Cristallographie- Thermodynamique, Faculté de Chimie, USTHB, BP 32 El-alia, Bab Ezzouar 16111, Alger, ⁽²⁾UMR 7036, Faculté des Sciences BP 239, 54506 Vandoeuvre-les-Nancy, France. E-mail: adjeghri@hotmail.com

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In our course of obtaining mixed oxides with specific properties by the polymeric precursors method, a novel barium biglutarate of formula Ba(C₅H₇O₄)₂ has been synthesized via silica medium synthetic route. Its X-ray single crystal structure has been determined at room temperature. It crystallizes in the space group P4₂/mbc with a=b=8.3460Å; c=18.5331Å and z=4. The single site for Ba²⁺ cation is the two-fold symmetry axis, while, there are two different sites for C and H atoms. One from three C atoms and three from five H atoms are located on the mirror plane, whereas all O atoms are in general positions. Each metal center is surrounded by eight ligands. The square antiprism coordination polyhedron is not very distorted owing to the single coordination mode within the hydrogeno glutarate group, giving μ-1.3- bridging. The noteworthy feature in the structure is the very short hydrogen bond bridging two oxygen atoms of carboxylate ends. The layer-type polymeric structure is built up from isolated BaO₈ polyhedra linked via conventional carboxylate bridges along [010] direction, and connected by the carbon backbone chains of the ligands along [001] direction. The resulting two-dimensional substructures form a pseudo-square grid network exhibiting wide empty channels parallel to [001] direction, giving a truly open framework.

The I.R spectra, consistent with crystallographic results, evidences shifting of the two characteristic stretching vibrations of COO group and shows sharp absorption of the strong H-bonded oxygen atoms.

The thermal analysis performed by ATG and DSC under N₂ or in air, reveal that the compound, stable up to 65°C, undergoes four significant endothermic peaks, and highlight the singular behavior of hydrogenoglutarate when compared to other glutarate or glutarate-biglutarate of earth-alkaline, and rare-earth metals. Further studies are still in progress in order to identify thermal intermediate and decomposition products and investigate their use as starting materials for obtaining interesting mixed oxides at low temperature.