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Polynuclear Coordination Polymer with Glutarate Ligand: $Sr(C_5H_7O_4)_2(H_2O)_2$

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In our course of obtaining new organic-inorganic complexes built up of flexible dicarboxylate ligands, we have noticed the peculiar behavior of the ligands derived from glutaric acid. Linked to rare-earth or alkaline-earth metals, they lead to a wide variety of structural types : catena polynuclear complex [1], uncommon cage feature [2], isolated polyhedra [3] involving completely or partially deprotonated unities. The new strontium biglutarate has been obtained on single crystal form via silica medium synthetic route. It is different from barium biglutarate in that it completes its coordination sphere by two coordinated water molecules, and crystalizes in space group P$\bar{1}$. The metal is nine coordinated forming infinite chains of one-antiprism $SrO_7(H_2O)_2$ within which te distance between two neighbouring Sr^{2+} ions are 4.272(6)Å. These chains are cross-linked, leading to a layered structure.

[1] Benmerad B., Guehria-Laïdoudi A., Balegroune F., Birkedal H., Chapuis G., *Acta cryst.*, 2000, **C56**, 789. [2] Benmerad B., Guehria-Laïdoudi A., Dahaoui S., Lecomte C., *Acta cryst.*, 2004, **C60**, m119. [3] Aliouane K., Djeghri A., Guehria-Laidoudi A., Dahaoui S., Lecomte C., *ECM-22*, Budapest, 2004.

Keywords: biglutarate, alcaline-earth complexes, organic-inorganic polymers

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Direct Observation of Photochemical Reactions by X-ray Crystallography – Supramolecular Approach

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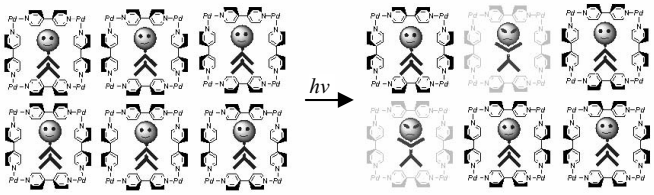
Kawano and coworkers crystallographically studied photo-induced reactive species such as radicals, carbenes, and nitrenes cryo-trapped in a crystal [1,2,3]. Here we propose a supramolecular approach for *in situ* observation to overcome practical problem of crystal deterioration by photo-irradiation as shown in scheme. We used a void in a self-assembled giant cage complex to control the reaction cavity. In this talk, we would like to introduce *in situ* observation of a photo-induced unsaturated transition metal complex and a crystalline state [2+2] reaction of acenaphtylene in a M6L4 cage [4].



[1] Kawano M., Sano T., Abe J., Ohashi Y., *J. Am. Chem. Soc.*, 1999, **121**, 8106. [2] Kawano M., Hirai K., Tomioka H., Ohashi Y., *J. Am. Chem. Soc.*, 2001, **123**, 6904. [3] Kawano M., Takayama T., Uekusa H., Ohashi Y., Ozawa Y., Matsubara K., Imabayashi H., Mitsumi M., Toriumi K., *Chem. Lett.*, 2003, 922. [4] Yoshizawa M., Takeyama Y., Kusukawa T., Fujita M., *Angew. Chem. Int. Ed.*, 2002, **41**, 1347.

Keywords: in situ observations, unstable compounds, photochemistry

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Metal-Ligand and Metal-Metal Bonding Characterization from X-ray Diffraction

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The metal complex of a non-steroidal anti-inflammatory drug (NSAID), di-μ-aspirinato-copper(II), crystallizes in its biochemical active form [1]. The electron density and electrostatic potential calculations are useful for the understanding of the metal-ligand bonding and its connection with the biochemical activity of such molecule. The compound also offers the opportunity to characterize the occurring copper-copper bond (Cu – Cu = 2.604 Å).

X-ray diffraction measurements were performed on a smart CCD diffractometer at 100 K with the MoKα radiation. up to sin θ/λ= 1.11 Å^{-1}. The compound crystallizes in the $P2_1/c$ group and reveals a one dimensional polymeric structure. The copper is in an irregular octahedral coordination corresponding to five oxygen atoms and one copper.

Electron density refinements are carried out using the Hansen-Coppens model [2]. The Cu-O, Cu-Cu electron density bonds are discussed according to the copper d-orbital populations. Electrostatic potential and active sites of the molecule are also shown.

[1] Garcia F., Méndez-Rojas M.A., González-Veraga E., Bernes S., Quiroz M.A., *Acta Cryst.*, 2003, **E59**, m1171-m1173. [2] Hansen N., Coppens P., *Acta Cryst*., 1978, **A34**, 909.

Keywords: electron density, electrostatic potential, metal-metal bonds

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Scanning Texture Analysis of Lamellar Bone using Microbeam Synchrotron Radiation

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Bone consists of mineral particle reinforced collagen and is structurally optimized for its biological functions. The 3D mineral nanostructure is still not fully understood. Specifically, little is known about the lamellar and sublamellar structure of the osteon, the fundamental unit of compact bone. The combination of microbeam (one micrometer) synchrotron scanning SAXS (small angle x-ray scattering) and WAXD (wide angle x-ray diffraction) allows us to reconstruct the full 3D mineral particle distribution at different positions within single osteonal lamellae. The WAXD data was used to calculate pole figures (stereographic projections), which delivers information on the mean orientation and texture of the principal axes of the mineral crystallites (which have a hexagonal cubic structure).

We scanned several osteons from a human femoral midshaft which were cut into slices of 3 micrometers. Our results show that the mineral crystal orientation has a fibre texture within single lamellae and shows intralamellar variation. The direction of mineral crystals rotates within the plane of each lamella.

Keywords: bone, texture analysis, synchrotron X-ray diffraction

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Palladium(II) and Platinum(II) Complexes with Tridentate Iminophosphine Ligands and their Phosphine Derivatives; Synthesis and Structural Studies

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