

**FA5-MS01-P01**

**Synthesis and Rietveld Refinements of Ca-Cu Hydroxyapatites.** M. Othmani Maseoud<sup>a</sup>, Abdallah Aissa<sup>a</sup>, Mongi Debbabi<sup>a</sup>. *Laboratory of Physico-Chemistry of Materials, Faculty of Science of Monastir, 5019 Monastir, Tunisia.*

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Solid solutions of calcium and copper hydroxyapatite were synthesized by precipitation method under specific conditions (pH=10, T=90°C) and their structural properties were investigated. Preliminary characterization by X-ray diffraction pattern showed that the solid solution Ca-CuHAp is limited to a general chemical formula:  $\text{Ca}_{(10-x)}\text{Cu}_x(\text{PO}_4)_6(\text{OH})_2$  with  $0 \leq x \leq 3,5$ . For  $x \geq 4$ , a new phase ( $\text{Cu}(\text{OH})\text{PO}_4 \cdot 0,2 \text{H}_2\text{O}$ ) is observed [1]. Chemical analysis showed that the amounts of copper contained in the samples are lower than those of synthetic solutions.

The IR spectroscopy analysis shows the presence of all vibrational bands characteristic of apatitic phase.

Structural refinement by the Rietveld method using Fullprof program indicates that solid solution is limited to  $x=1,23$ . The replacement of calcium by copper in hydroxyapatite structure leads to the decrease of lattice parameters a and c according to Vegard's law. The distribution of copper between the two crystallographic sites S(I) and S(II) of the apatitic structure is close to the statistical distribution, with a slight preference to the sites S(I).

[1] V. Kopylovich, L. Shchegrov, Yu. Vovkotrub ; *Russ. J. Inorg. Chem.* **1993**, 38, 1018.

**Keywords:** hydroxyapatite; rietveld method; crystallographic sites

**FA5-MS01-P02**

**Testing Different Methods for Crystal Structure Determination of Alaptide from Powder Diffraction Data.** Jan Rohlíček<sup>a</sup>, Michal Hušák<sup>a</sup>, Bohumil Kratochvíl<sup>a</sup>. *Department of Solid State Chemistry, Institute of Chemical-Technology in Prague, Czech Republic.*

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Crystal structure determination of small organic molecules from X-Ray powder diffraction data is going to be a routine method. There exist several methods how to obtain the result so we had for one compound tested and compared multiple ones.

We choose alaptide and we tried to solve the crystal structure of this small pharmaceutically relevant molecule by using different methods. Preliminary we measured a powder pattern on a laboratory diffractometer. Then we used quantum mechanics computation combined with lattice simulation, so we tried to predict this structure. The measured pattern serves just for choosing the correct result from the suggested structures list. The second way how to solve a crystal structure of alaptide was solving the structure from laboratory and synchrotron X-Ray powder diffraction

data by using both direct methods and direct space methods. We choose parallel tempering algorithm as implemented in the program FOX [1] (direct space methods) and we tested program Expo2004 [2] (direct method). Finally we made refinement of all results and we compared results of all used methods together.

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[1] V. Favre-Nicolin and R. Cerny, *J. Appl. Cryst.* **35**, **2002**, 734-743. [2] Altomare, A., Giacovazzo, C., Guagliardi, A., Moliterni, A.G.G., Rizzi, R., & P.-E. Werner, **2000**. *J. Appl. Cryst.* **33**, 1180-1186

**Keywords:** ab-initio structure determination; powder structure determination; powder X-ray diffraction

**FA5-MS01-P03**

**Two New Isotypes Arsenate Compounds of Transition Metals:  $\text{A}_3\text{Nb}_3\text{WAs}_2\text{O}_{15}$  (A:Rb,Cs).** Ratiba Belhouas<sup>a</sup>, Djillali Mezaoui<sup>a</sup>, Houria Rebbah<sup>a</sup>. *LABORATOIRE DES SCIENCES DES MATÉRIEAUX (LABSCIMAT) FACULTÉ DE CHIMIE, UNIVERSITÉ DES SCIENCES ET DE LA TECHNOLOGIE HOUARI BOUMEDIENE (U.S.TH.B), BP N°32, EL-ALIA, ALGIERS, ALGERIA.*

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The arsenates transition metals exhibit interesting conduction, ionic exchange and catalytic properties [1, 2]. In this study we present two new isotypes of arsenate compounds.

These new compounds were synthesized by solid state reaction and characterized by x-rays fluorescence (XRF), powder diffraction (DRX) as well as a measure of its density by pycnometry.

The determination of its crystallographic characteristic, its evolution and the influence of this total substitution on the stability of the structure will be discussed.

We are interested to the arsenate of potassium  $\text{K}_3\text{Nb}_3\text{WAs}_2\text{O}_{15}$  [3] isostructural to the phosphate of potassium of general composition  $\text{K}_3\text{Nb}_3\text{WP}_2\text{O}_{17}$  with mixed frameworks structure [4]. In order to extend this arsenate compound family, a total cationic replacement in the alkaline cation in insertion by the rubidium and cesium was realized and lead us to the title isotype compounds  $\text{A}_3\text{Nb}_3\text{WAs}_2\text{O}_{15}$  (A : Rb, Cs).

[1] Winand J.M., Rulment. A. & Tartre, P., *J of Solid State Chem*, **1990**, 87, 83. [2] Bestaoui. N., Verbaère, Piffard. Y., Coulibaly. V. & Zah Letho, *J. Eur. J. Solid. State. Inorg. Chem.*, **2002**, 169, 160. [3] Belkhiri S, Ouhenia. S, Mezaoui. D., Belkhiri M.A. & Rebbah. H., *Powder. Diffr.*, **2006**, 21, 3. [4] Berrah H., Mezaoui D., Guesdon A., Borel M.M, Leclair A., Provost J. & Raveau B., *Chem. Mater.*, **1998**, 10, 543.

**Keywords:** arsenate; cationic substitution; solid state synthesis